Bimetallic Coordination Insertion Polymerization of Unprotected Polar Monomers: Copolymerization of Amino Olefins and Ethylene by Dinickel Bisphenoxyiminato Catalysts

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Supporting Online Material

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Polymerization Tables

Table S1. Ethylene/1-hexene copolymerizations with TON of ethylene and 1-hexene.[a]

	complex	additive	yield (g)	activity[b]	R[c]	% incorp[d]	TON ^{ethylene}	TON $^{1\text{-hexene}}$
1	1-s-Py	none[e]	0.003	1		--	--	--
2	1-s-Py	none[f]	0.033	1		1.0	48	0.5
3	1-s	none[e]	1.266	317		1.2	10900	135
4	1-a	none[e]	1.590	397		1.1	13700	161
5	2-s	none[e]	1.444	361		1.1	12400	148
6	2-a	none[e]	1.469	367		1.0	12700	136
7	2-s-OMe	none[e]	1.095	274		0.9	9490	88
8	1-s	$NMeEt_2$	0.187	23	13	0.8	815	6.6
9	1-a	$NMeEt_2$	0.106	13	30	0.5	465	2.3
10	2-s	$NMeEt_2$	0.199	25	15	0.3	878	3.0
11	2-a	$NMeEt_2$	0.087	11	34	0.9	378	3.4
12	2-s-OMe	$NMeEt_2$	0.142	18	17	0.7	618	4.4
13	1-s	NEt_3	0.073	9	33	1.0	315	3.0
14	1-a	NEt_3	0.011	1	290	1.3	46	0.6
15	2-s	NEt_3	0.447	56	7	0.3	1980	5.3
16	2-a	NEt_3	0.006	1	515	--	--	--
17	2-s-OMe	NEt_3	0.457	57	5	0.7	1990	15
18	1-s	NMe^nPr_2	0.094	12	26	0.9	407	3.7
19	1-a	NMe^nPr_2	0.005	1	639	1.2	21	0.3
20	2-s	NMe^nPr_2	0.783	98	4	0.2	3460	8.2
21	2-a	NMe^nPr_2	--[g]	--	--	--	--	--
22	2-s-OMe	NMe^nPr_2	0.165	21	14	0.7	718	5.1
23	1-s	N^nPr_3	--[g]	--	--	--	--	--
24	1-a	N^nPr_3	--[g]	--	--	--	--	--
25	2-s	N^nPr_3	0.447	56	6	0.2	1980	3.4
26	2-a	N^nPr_3	--[g]	--	--	--	--	--
27	2-s-OMe	N^nPr_3	0.390	49	6	0.5	1720	7.9

[a]All polymerizations were run for 1 h at 25°C under 100 psig of ethylene in toluene with 4 μmol of dinickel complex, 4 equivalents of $Ni(COD)_2$ and 500 equivalents of 1-hexene and additive per nickel. Reaction total volume = 5 mL. [b]activity = (g polymer)×(mmol Ni)$^{-1}$×h^{-1}. [c]R = [activity with no additive]/[activity with additive]. [d]% incorp = mole percent incorporation of 1-hexene as determined from ^{1}H and ^{13}C NMR spectroscopy. [e]Polymerization run for 0.5 h due to high viscosity limiting mass transfer by that time. [f]Polymerization run for 3 h to get enough polymer for ^{1}H and ^{13}C NMR spectra. [g]Insufficient product to accurately mass (<1 mg).

Table S2. Ethylene/amino olefin copolymerizations with TON of ethylene and amino olefin.[a]

	complex	comonomer	yield (g)	activity[b]	% incorp[c]	TON ^{ethylene}	TON $^{amino\ olefin}$
1	1-s	$N(allyl)^nPr_2$	0.034	8	0.0	150	--
2	2-s	$N(allyl)^nPr_2$	0.083	21	0.1	366	0.3
3	1-s	$N(butenyl)^nPr_2$	0.019	5	0.4	84	0.4
4	2-s	$N(butenyl)^nPr_2$	0.044	11	0.4	192	0.7
5	1-s	$N(pentenyl)^nPr_2$	0.064	16	0.5	273	1.4
6	2-s	$N(pentenyl)^nPr_2$	0.178	45	0.3	780	2.5
7	1-s	$N(hexenyl)^nPr_2$	0.063	16	0.7	270	1.8
8	2-s	$N(hexenyl)^nPr_2$	0.217	54	0.3	945	3.3
9	1-s	$N(heptenyl)^nPr_2$	0.059	15	0.8	250	2.0
10	2-s	$N(heptenyl)^nPr_2$	0.310	78	0.3	1360	3.8
11	1-s	$N(octenyl)^nPr_2$	0.050	13	0.7	213	1.5
12	2-s	$N(octenyl)^nPr_2$	0.398	100	0.3	1740	4.7
13	1-s	$N(pentenyl)Et_2$	0.034	8	0.5	146	0.7
14	2-s	$N(pentenyl)Et_2$	0.151	38	0.3	665	1.7

15	1-a	N(pentenyl)^{n}Pr$_2$	--[d]	--[d]	--[d]	--[d]	--[d]
16	2-a	N(pentenyl)^{n}Pr$_2$	0.032	8	0.7	137	0.9
17	2-s-OMe	N(pentenyl)^{n}Pr$_2$	0.176	44	0.3	769	2.5

[a]All polymerizations were run for 0.5 h at 25°C under 100 psig of ethylene in toluene with 4 μmol of dinickel complex, 4 equivalents of Ni(COD)$_2$ and 500 equivalents of comonomer per nickel. Reaction total volume = 5 mL. [b]activity = (g polymer)×(mmol Ni)$^{-1}$×h^{-1}. [c]% incorp = mole percent incorporation of comonomer as determined from ^{1}H NMR spectroscopy. [d]Insufficient product to accurately mass (<1 mg).

Table S3. Ethylene/1-hexene copolymerizations run for different lengths of time.[a]

	complex	additive	time (minutes)	yield (g)	activity[b]
1	1-s	none	30	1.266	317
2	1-s	none	30	1.156	289
3	1-a	none	30	1.590	397
4	1-a	none	30	1.540	385
5	2-s	none	30	1.444	361
6	2-s	none	30	1.461	366
7	2-a	none	30	1.469	367
8	2-a	none	30	1.464	366
9	2-s-OMe	none	30	1.095	274
10	2-s-OMe	none	30	1.262	315
11	1-s	NMe^{n}Pr$_2$	60	0.094	12
12	1-s	NMe^{n}Pr$_2$	60	0.064	8
13	1-s	NMe^{n}Pr$_2$	30	0.023	6
14	1-s	NMe^{n}Pr$_2$	30	0.044	11
15	1-s	NMe^{n}Pr$_2$	5	0.002	3
16	1-s	NMe^{n}Pr$_2$	5	0.002	3
17	2-s	NMe^{n}Pr$_2$	60	0.783	98
18	2-s	NMe^{n}Pr$_2$	30	0.335	84
19	1-a	NEt$_3$	60	0.011	1
20	1-a	NEt$_3$	30	0.002	1
21	2-a	NEt$_3$	60	0.006	1
22	2-a	NEt$_3$	30	--[c]	--[c]

[a]All polymerizations were run at 25°C under 100 psig of ethylene in toluene with 4 μmol of dinickel complex, 4 equivalents of Ni(COD)$_2$ and 500 equivalents of 1-hexene and additive per nickel. Reaction total volume = 5 mL. [b]activity = (g polymer)×(mmol Ni)$^{-1}$×h^{-1}. [c]Insufficient product to accurately mass (<1 mg).

Table S4. Ethylene/amino olefin copolymerizations run for different lengths of time.[a]

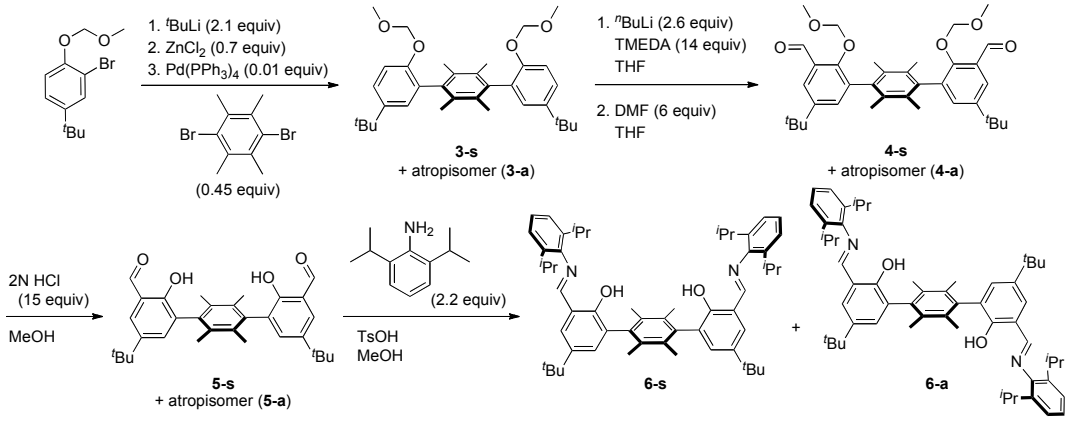
	complex	comonomer	time (minutes)	yield (g)	activity[b]	% incorp[c]	TON^{ethylene}	TON$^{amino\ olefin}$
1	1-s	N(pentenyl)^{n}Pr$_2$	240	0.063	2	n.d.	n.d.	n.d.
2	1-s	N(pentenyl)^{n}Pr$_2$	60	0.067	8	0.54	286	1.6
3	1-s	N(pentenyl)^{n}Pr$_2$	30	0.064	16	0.5	273	1.4
4	1-s	N(pentenyl)^{n}Pr$_2$	5	0.054	81	0.82	231	1.9
5	2-s	N(pentenyl)^{n}Pr$_2$	30	0.178	45	0.3	780	2.5
6	2-s	N(pentenyl)^{n}Pr$_2$	30	0.240	60	0.3	1050	2.8
7	2-s	N(pentenyl)^{n}Pr$_2$	5	0.085	128	0.3	370	1.3
8	2-a	N(pentenyl)^{n}Pr$_2$	30	0.032	8	0.7	137	0.9
9	2-a	N(pentenyl)^{n}Pr$_2$	30	0.034	8	0.7	143	1.0
10	2-a	N(pentenyl)^{n}Pr$_2$	5	0.004	7	0.6	17	0.1

[a]All polymerizations were run at 25°C under 100 psig of ethylene in toluene with 4 μmol of dinickel complex, 4 equivalents of Ni(COD)$_2$ and 500 equivalents of comonomer per nickel. Reaction total volume = 5 mL. [b]activity = (g polymer)×(mmol Ni)$^{-1}$×h^{-1}. [c]% incorp = mole percent incorporation of comonomer as determined from ^{1}H NMR spectroscopy. n.d. = not determined [d]Insufficient product to accurately mass (<1 mg).

Experimental Section

General Considerations and Instrumentation. All air- and/or water-sensitive compounds were manipulated using standard vacuum or Schlenk line techniques or in an inert atmosphere glove box. The solvents for air- and moisture-sensitive reactions were dried over sodium benzophenone ketyl, calcium hydride, or by the method of Grubbs.[1] All NMR solvents were purchased from Cambridge Isotopes Laboratories, Inc. Benzene-d_6 was dried over sodium benzophenone ketyl and vacuum transferred prior to use. Pyridine and all monomers and amines were dried over calcium hydride and vacuum transferred prior to use. Trimethylphosphine was dried over 4 Å molecular sieves prior to use. Ethylene was purchased from Matheson and equipped with a PUR-Gas in line trap to remove oxygen and moisture before use. All ^{1}H, ^{13}C, ^{31}P, and 2D NMR spectra of small organic and organometallic compounds were recorded on Varian Mercury 300 MHz, Varian 400 MHz, or Varian INOVA-500 or 600 MHz spectrometers at room temperature. All ^{1}H and ^{13}C NMR spectra of polymers were recorded on the Varian INOVA-500 MHz spectrometer at 130 °C in tetrachloroethane-d_2. For ^{1}H and ^{13}C NMR spectra, chemical shifts are reported with respect to residual internal protio solvent: 7.16 and 128.06 (t) ppm (C_6D_6); 7.26 and 77.16 (t) ppm ($CDCl_3$); 5.32 and 53.84 (q) ppm (CD_2Cl_2); for ^{1}H and ^{13}C data. All ^{31}P NMR spectra were externally referenced to H_3PO_4 (0 ppm). Combustion analyses (C, H, and N) were performed by Complete Analysis Laboratories, Inc., Parsippany, NJ. **1-s-Py** was synthesized according to literature procedure.[2]

Preparation of ligands



Scheme S1. Synthesis of para-bisphenoxyiminato compounds (alternative to previously reported route).[2]

2-bromo-4-*tert*-butylphenol,[3] 2-bromo-4-*tert*-butylmethoxymethylphenol,[2] and 1,4-dibromo-2,3,5,6-tetramethylbenzene[4] were synthesized according to literature procedures.

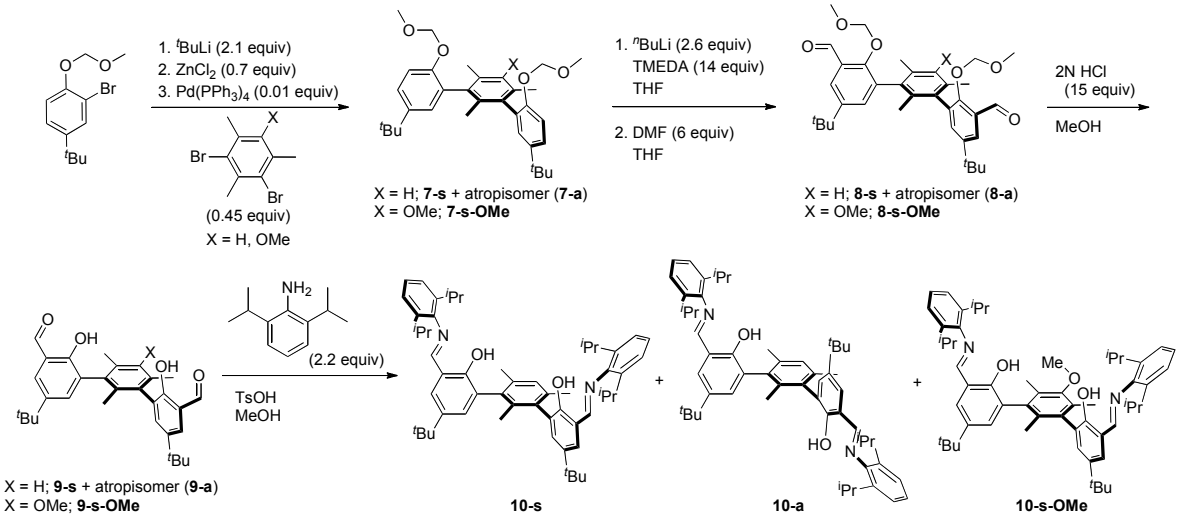
3-a and **3-s**. Synthesis of the biphenyl compounds **3** was accomplished via the Negishi coupling of 1,4-dibromo-2,3,5,6-tetramethybenzene with two equivalents of 2-bromo-4-*tert*-butylmethoxymethylphenol using literature conditions.[5] In the glove box, 2-bromo-4-*tert*-butylmethoxymethylphenol (10.46 g, 38.3 mmol) and 150 mL of THF were combined in a large Schlenk tube and frozen in the cold well. *tert*-Butyllithium (47.32 mL, 80.44 mmol, 2.1 equiv) was added to the thawing solution and stirred for 1 h while warming to room temperature. The resultant yellow orange solution was refrozen in the cold well. Concurrently, a suspension of $ZnCl_2$ (3.66 g, 26.8 mmol, 0.7 equiv) in THF (40 mL) was frozen in the cold well. The thawing

$ZnCl_2$ suspension was added to the thawing reaction mixture and stirred for 1 h resulting in a colorless cloudy solution. 1,4-dibromo-2,3,5,6-tetramethybenzene (5.03 g, 17.24 mmol, 0.45 equiv), $Pd(PPh_3)_4$ (0.44 g, 0.38 mmol, 0.01 equiv) and THF (40 mL) were added to the reaction mixture at room temperature. The sealed Schlenk tube was brought out of the glove box and heated to 70 °C for 4 days. Water was added to quench the reaction. The solution was filtered over silica gel and the silica gel was washed with dichloromethane. The filtrate was extracted between DCM and water. The organics were dried with $MgSO_4$, filtered, and volatiles were removed under vacuum. The dicoupled products were coprecipitated from MeOH in a ratio of 1:0.73 anti:syn (4.75 g of white powder, 53 % yield) and used without further purification. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.33-7.29 (2dd, *J*=8.6 Hz, 2.5, 2H per atropisomer, Ar*H*), 7.17-7.10 (4d, *J*=8.6 Hz, 2.5, 4H per atropisomer, Ar*H*), 5.07 (s, 4H, OCH_2OCH_3, anti), 4.97 (s, 4H, OCH_2OCH_3, syn), 3.37 (s, 6H, OCH_2OCH_3, anti), 3.24 (s, 6H, OCH_2OCH_3, syn), 1.97 (s, 12H, Ar-CH_3, syn), 1.95 (s, 12H, Ar-CH_3, anti), 1.32 (s, 18H per atropisomer, $ArC(CH_3)_3$) ppm.

4-a and **4-s**. **3** (4.75 g, 9.16 mmol, 1 equiv), *N,N,N',N'*-tetramethylethylenediamine (19.1 mL, 128.2 mmol, 14 equiv) and THF (80 mL) were added to a Schlenk tube in the glove box and frozen in the cold well. *n*-Butyllithium (9.6 mL, 24.0 mmol, 2.6 equiv) was added to the thawing solution and stirred for 4 h. The resultant orange red solution was refrozen in the cold well. A solution of DMF (4.3 mL, 54.9 mmol, 6 equiv) in THF (30 mL) was also frozen in the cold well. The thawing DMF solution was added to the thawing reaction mixture resulting in a pale amber solution, which was stirred for 10 h before the Schlenk tube was brought out of the box and about 5 mL of water were added to quench the reaction. The desired product was extracted into DCM and the organic fraction was washed with water, dried with $MgSO_4$, filtered, and the volatiles were removed under vacuum to yield the doubly orthoformylated products, **4**, with greater than 90% purity. These compounds were carried forward without further purification and 100% conversion was assumed for stoichiometry. ^{1}H NMR (300 MHz, $CDCl_3$): δ 10.49 (2s, 2H per atropisomer, O*CH*), 7.90 (2d, *J*=2.7 Hz, 2H per atropisomer, Ar*H*), 7.43 (d, *J*=2.7 Hz, 2H, Ar*H*, syn), 7.31 (d, *J*=2.6 Hz, 2H, Ar*H*, anti), 4.67 (s, 4H, OCH_2OCH_3, anti), 4.61 (s, 4H, OCH_2OCH_3, syn), 3.29 (s, 6H, OCH_2OCH_3, syn), 3.24 (s, 6H, OCH_2OCH_3, anti), 2.03 (s, 12H, Ar-CH_3, syn), 2.00 (s, 12H, Ar-CH_3, anti), 1.35 (2s, 18H per atropisomer, $ArC(CH_3)_3$) ppm.

5-a and **5-s**. **4** (5.26 g, 9.2 mmol, 1 equiv), 2N HCl (69 mL, 137.4 mmol, 15 equiv), and methanol (275 mL) were added to a round bottom flask equipped with a reflux condenser and the reaction was refluxed for 2 h. Then the reaction was cooled to room temperature and filtered to collect precipitate. The precipitate was dissolved in DCM and washed with water. Additional product was collected from the filtrate by extraction into DCM. The organic fractions were dried with $MgSO_4$, filtered, and the volatiles were removed under vacuum to yield the deprotected products, **5**. The atropisomers were separated via column chromatography (3:1 DCM/hexanes). 1.3 g of **5-a** and 0.7 g of **5-s** were isolated as white solids in 95 % puritiy (45 % overall yield). The ^{1}H and ^{13}C NMR spectra matched literature assignments.[2]

6-a and **6-s**. The bisphenoxyimines were synthesized as we described previously from the bisaldehydes from **5-a** and **5-s**.[2]



Scheme S2. Synthesis of meta-bisphenoxyiminato compounds.[2]

1,3-dibromomesitylene was synthesized according to literature procedures.[6]

3,5-dibromomesitol was synthesized by the dropwise addition of a solution of Br_2 (1.48 mL, 28.76 mmol, 2.61 equiv) in DCM (6 mL) through an addition funnel to a solution of mesitol (1.5 g, 11.01 mmol, 1 equiv) and iron powder (0.04 g, 0.71 mmol, 0.06 equiv) in DCM (11 ml) in a covered round bottom flask with a stirbar over 30 minutes. The resulting red solution was sitrred an additional 2.5 h before the reaction was quenched with aqueous sodium hydrosulfite ($Na_2S_2O_4$). The mixture was extracted between DCM and water. The organics were dried with $MgSO_4$, filtered, and volatiles were removed under vacuum. The desired product was obtained as a pale yellow solid in quantitative yield. The ^{1}H NMR spectrum matched literature assignments.[7]

3,5-dibromo-2,4,6-trimethyl-methoxybenzene was synthesized from 3,5-dibromomesitol according to an analogous synthesis.[8] ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.67 (s, 3H, OCH_3), 2.61 (s, 3H, ArCH_3), 2.36 (s, 6H ArCH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 155.28 (Ar), 133.59 (Ar), 130.98 (Ar), 125.56 (Ar), 60.68 (ArO*C*H$_3$), 25.29 (Ar*C*H$_3$), 17.61 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{10}H_{12}OBr^{81}Br$: 307.9234. Found: 307.9246.

7-a and **7-s**. Synthesis of the biphenyl compounds **7** was accomplished via the Negishi coupling of 1,3-dibromomesitylene with two equivalents of 2-bromo-4-*tert*-butylmethoxymethylphenol using conditions analogous to the synthesis of compounds **3**. Precipitation from methanol yielded **7-a** as a white solid (2.5 g). **7-s** was collected in greater than 80 % purity via column chromatography (30:1 hexanes/ethyl acetate). **7-s** was then isolated as a colorless oil (2.3 g) via column chromatography of the 3:2 hexanes/ethyl acetate flush of the previous column (6.25:1 hexanes/ethyl acetate) (58 % total yield of both atropisomers). **7-a** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.31 (dd, *J*=8.6, 2.6 Hz, 2H, Ar*H*), 7.16 (d, *J*=8.6 Hz, 2H, Ar*H*), 7.11 (d, *J*=2.5 Hz, 2H, Ar*H*), 7.09 (s, 1H, Ar*H*), 5.13 (d, *J*=6.8 Hz, 2H, OCH_2OCH$_3$), 5.08 (d, *J*=6.8 Hz, 2H, OCH_2OCH$_3$), 3.36 (s, 6H, OCH$_2$OCH_3), 2.08 (s, 6H, ArCH_3), 1.72 (s, 3H, ArCH_3), 1.30 (s, 18H, ArC(CH_3)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 152.29 (Ar), 144.76 (Ar), 136.50 (Ar), 135.51 (Ar), 135.50 (Ar), 130.41 (Ar), 128.67 (Ar), 128.42 (Ar), 124.77 (Ar), 114.31 (Ar), 94.63 (O*C*H$_2$OCH$_3$), 55.90 (OCH$_2$O*C*H$_3$), 34.34 (Ar*C*(CH$_3$)$_3$), 31.66 (ArC(*C*H$_3$)$_3$), 20.82 (Ar*C*H$_3$), 18.49 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{33}H_{44}O_4$: 504.3240. Found: 504.3251. **7-s** ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 (dd, *J*=8.6, 2.6 Hz, 2H, Ar*H*), 7.19 (d, *J*=2.6 Hz, 2H, Ar*H*), 7.15 (d, *J*=8.6 Hz, 2H, Ar*H*), 7.11 (s, 1H, Ar*H*), 5.05 (d, *J*=6.6 Hz, 2H, OCH_2OCH$_3$), 5.01 (d, *J*=6.6 Hz, 2H, OCH_2OCH$_3$), 3.32 (s, 6H, OCH$_2$OCH_3),

2.09 (s, 6H, ArCH_3), 1.75 (s, 3H, ArCH_3), 1.33 (s, 18H, ArC(CH_3)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 152.12 (Ar), 145.09 (Ar), 136.51 (Ar), 135.62 (Ar), 135.56 (Ar), 130.74 (Ar), 128.60 (Ar), 128.46 (Ar), 124.81 (Ar), 115.03 (Ar), 94.92 (O*C*H$_2$OCH$_3$), 55.82 (OCH$_2$O*C*H$_3$), 34.40 (Ar*C*(CH$_3$)$_3$), 31.68 (ArC(*C*H$_3$)$_3$), 20.83 (Ar*C*H$_3$), 18.52 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{33}H_{44}O_4$: 504.3240. Found: 504.3228.

7-s-OMe. Synthesis of the biphenyl compound **7-s-OMe** was accomplished via the Negishi coupling of 3,5-dibromo-2,4,6-trimethyl-methoxybenzene with two equivalents of 2-bromo-4-*tert*-butylmethoxymethylphenol using conditions analogous to the synthesis of compounds **3**. **7-s-OMe** was collected in greater than 90 % purity via column chromatography (5:1:0.5 hexanes/ethyl acetate/DCM). **7-s-OMe** was then isolated as a colorless solid via precipitation from MeOH (4.0 g, 16 % yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.31 (dd, *J*=8.6, 2.5 Hz, 2H, Ar*H*), 7.17 (d, *J*=2.5 Hz, 2H, Ar*H*), 7.11 (d, *J*=8.6 Hz, 2H, Ar*H*), 5.00 (d, *J*=6.5 Hz, 2H, OC*H*$_2$OCH$_3$), 4.97 (d, *J*=6.5 Hz, 2H, OC*H*$_2$OCH$_3$), 3.78 (s, 3H, ArOC*H*$_3$), 3.28 (s, 6H, OCH$_2$OC*H*$_3$), 2.04 (s, 6H, ArC*H*$_3$), 1.68 (s, 3H, ArC*H*$_3$), 1.31 (s, 18H, ArC(C*H*$_3$)$_3$) ppm. ^{13}C NMR (75 MHz, C_6D_6): δ 155.53 (Ar), 152.84 (Ar), 145.11 (Ar), 138.51 (Ar), 131.65 (Ar), 131.07 (Ar), 128.84 (Ar), 128.56 (Ar), 125.33 (Ar), 115.84 (Ar), 95.02 (O*C*H$_2$OCH$_3$), 59.60 (O*C*H$_3$), 55.55 (O*C*H$_3$), 34.27 (Ar*C*(CH$_3$)$_3$), 31.66 (ArC(*C*H$_3$)$_3$), 18.94 (Ar*C*H$_3$), 14.47 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{34}H_{46}O_5$: 534.3345. Found: 534.3346.

8-a. Formylation of **7-a** was accomplished via the same procedure as for **4**. 0.53 g of doubly orthoformylated compound was collected as a pale yellow solid after extraction and moved forward without further purification (53 % yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.44 (s, 2H, C*H*O), 7.88 (d, J = 2.6 Hz, 2H, Ar*H*), 7.33 (d, J = 2.6 Hz, 2H, Ar*H*), 7.09 (s, 1H, Ar*H*), 4.73 (d, J = 6.0 Hz, 2H, OC*H*$_2$OCH$_3$), 4.70 (d, J = 6.0 Hz, 2H, OC*H*$_2$OCH$_3$), 3.21 (s, 6H, OCH$_2$OC*H*$_3$), 2.09 (s, 6H, ArC*H*$_3$), 1.81 (s, 3H, ArC*H*$_3$), 1.34 (s, 18H, ArC(C*H*$_3$)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 191.05 (*C*HO), 155.38 (Ar), 147.78 (Ar), 136.22 (Ar), 135.45 (Ar), 135.41 (Ar), 135.39 (Ar), 134.89 (Ar), 129.47 (Ar), 129.30 (Ar), 124.62 (Ar), 99.84 (O*C*H$_2$OCH$_3$), 57.30 (OCH$_2$O*C*H$_3$), 34.71 (Ar*C*(CH$_3$)$_3$), 31.38 (ArC(*C*H$_3$)$_3$), 20.91 (Ar*C*H$_3$), 18.85 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{35}H_{44}O_6$: 560.3138. Found: 560.3150.

8-s. Formylation of **7-s** was accomplished via the same procedure as for the anti, but a mixture of species resulted. Column chromatography was used to isolate the desired product as a pale yellow solid (0.30 g, 51 % yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.49 (s, 2H, C*H*O), 7.89 (d, J = 2.6 Hz, 2H, Ar*H*), 7.45 (d, J = 2.6 Hz, 2H, Ar*H*), 7.15 (s, 1H, Ar*H*), 4.65 (s, 4H, OC*H*$_2$OCH$_3$), 3.36 (s, 6H, OCH$_2$OC*H*$_3$), 2.12 (s, 6H, ArC*H*$_3$), 1.80 (s, 3H, ArC*H*$_3$), 1.34 (s, 18H, ArC(C*H*$_3$)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 191.19 (*C*HO), 155.40 (Ar), 147.87 (Ar), 136.50 (Ar), 135.63 (Ar), 135.58 (Ar), 134.32 (Ar), 129.61 (Ar), 129.58 (Ar), 124.21 (Ar), 99.34 (O*C*H$_2$OCH$_3$), 57.76 (OCH$_2$O*C*H$_3$), 34.81 (Ar*C*(CH$_3$)$_3$), 31.40 (ArC(*C*H$_3$)$_3$), 20.93 (Ar*C*H$_3$), 18.72 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{35}H_{44}O_6$: 560.3138. Found: 560.3151.

8-s-OMe. Formylation of **7-s-OMe** was accomplished via the same procedure as for **4**. 4.48 g of about 80% pure doubly orthoformylated compound was collected as a pale yellow solid after extraction and running through a silica plug to remove brown color with 3:1 hexanes/ethyl acetate. This material was moved forward without further purification. ^{1}H NMR (300 MHz, $CDCl_3$): δ 10.48 (s, 2H, C*H*O), 7.90 (d, J = 2.6 Hz, 2H, Ar*H*), 7.44 (d, J = 2.6 Hz, 2H, Ar*H*), 4.64 (s, 2H, OC*H*$_2$OCH$_3$), 3.78 (s, 3H, ArOC*H*$_3$), 3.35 (s, 3H, OCH$_2$OC*H*$_3$), 2.09 (s, 6H, ArC*H*$_3$), 1.75 (s, 3H, ArC*H*$_3$), 1.34 (s, 18H, ArC(C*H*$_3$)$_3$) ppm.

9-a. Deprotection of **8-a** was accomplished via the same procedure as for **5**. The desired product was isolated as an olive green solid in 95 % yield (0.86 g) after extraction. ^{1}H NMR (500 MHz, $CDCl_3$): δ 11.06 (d, J = 0.5 Hz, 2H, ArO*H*), 9.96 (s, 2H, C*H*O), 7.54 (d, J = 2.5 Hz,

2H, Ar*H*), 7.52 (dd, J = 2.5, 0.5 Hz, 2H, Ar*H*), 7.14 (s, 1H, Ar*H*), 2.07 (s, 6H, ArCH_3), 1.75 (s, 3H, ArCH_3), 1.34 (s, 18H, ArC(CH_3)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 197.12 (*C*HO), 156.79 (Ar), 143.01 (Ar), 137.00 (Ar), 136.46 (Ar), 135.52 (Ar), 134.02 (Ar), 129.69 (Ar), 129.22 (Ar), 128.99 (Ar), 120.17 (Ar), 34.40 (Ar*C*(CH$_3$)$_3$), 31.42 (ArC(*C*H$_3$)$_3$), 20.69 (Ar*C*H$_3$), 18.28 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{31}H_{36}O_4$: 472.2614. Found: 472.2593.

9-s. Deprotection of **8-s** was accomplished via the same procedure as for the anti. The desired product was isolated as a brown solid in quantitative yield (0.69 g) after extraction. ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.94 (s, 2H, ArO*H*), 9.96 (s, 2H, C*H*O), 7.54 (d, J = 2.5 Hz, 2H, Ar*H*), 7.48 (d, J = 2.5 Hz, 2H, Ar*H*), 7.15 (s, 1H, Ar*H*), 2.08 (s, 6H, ArCH_3), 1.75 (s, 3H, ArCH_3), 1.35 (s, 18H, ArC(CH_3)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 196.89 (*C*HO), 156.96 (Ar), 142.71 (Ar), 136.50 (Ar), 136.47 (Ar), 135.92 (Ar), 134.26 (Ar), 129.64 (Ar), 129.13 (Ar), 128.97 (Ar), 120.27 (Ar), 34.37 (Ar*C*(CH$_3$)$_3$), 31.44 (ArC(*C*H$_3$)$_3$), 20.74 (Ar*C*H$_3$), 18.21 (Ar*C*H$_3$) ppm. HRMS (FAB+) Calcd. for $C_{31}H_{37}O_4$: 473.2692. Found: 473.2675.

9-s-OMe. Deprotection of **8-s-OMe** was accomplished via the same procedure as for **5**. The desired product was isolated as beige solid in 70 % yield (2.66 g) after extraction and column chromatography (10:1 hexanes/ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.96 (s, 2H, ArO*H*), 9.96 (s, 2H, C*H*O), 7.55 (d, J = 2.5 Hz, 2H, Ar*H*), 7.47 (d, J = 2.5 Hz, 2H, Ar*H*), 3.79 (s, 3H, ArOCH_3), 2.05 (s, 6H, ArCH_3), 1.70 (s, 3H, ArCH_3), 1.36 (s, 18H, ArC(CH_3)$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 196.89 (*C*HO), 156.93 (Ar), 154.92 (Ar), 142.72 (Ar), 136.51 (Ar), 135.59 (Ar), 131.42 (Ar), 129.70 (Ar), 129.62 (Ar), 129.03 (Ar), 120.28 (Ar), 60.17 (ArO*C*H$_3$) 34.37 (Ar*C*(CH$_3$)$_3$), 31.44 (ArC(*C*H$_3$)$_3$), 18.13 (Ar*C*H$_3$), 14.08 (Ar*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{32}H_{38}O_5$: 502.2719. Found: 502.2718.

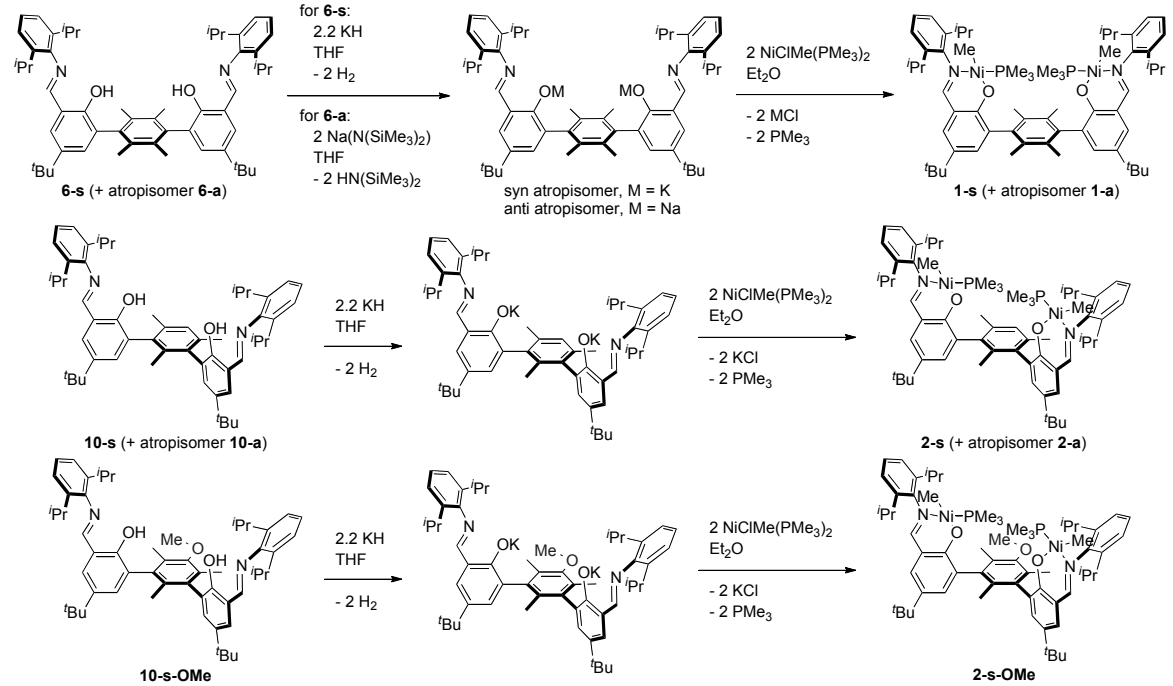
10-a. The imine condensation with 2,6-diisopropylaniline and **9-a** was accomplished via the same procedure as for **6-a**. The desired product was isolated as a pale yellow solid in 66 % yield (0.26 g). ^{1}H NMR (500 MHz, $CDCl_3$): δ 13.13 (s, 2H, ArO*H*), 8.38 (s, 2H, NC*H*) 7.45 (d, J = 2.4 Hz, 2H, Ar*H*), 7.32 (d, J = 2.4 Hz, 2H, Ar*H*), 7.20 (m, 7H, Ar*H*), 3.07 (hept, J = 6.8 Hz, 4H, ArC*H*(CH$_3$)$_2$), 2.18 (s, 6H, ArCH_3), 1.97 (s, 3H, ArCH_3), 1.36 (s, 18H, ArC(CH_3)$_3$), 1.20 (d, J = 6.8 Hz, 12H, ArCH(CH_3)$_2$), 1.18 (d, J = 6.8 Hz, 12H, ArCH(CH_3)$_2$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 167.16 (N*C*H), 156.41 (Ar), 146.55 (Ar), 141.65 (Ar), 138.92 (Ar), 136.15 (Ar), 135.69 (Ar), 135.04 (Ar), 133.31 (Ar), 129.52 (Ar), 129.22 (Ar), 127.32 (Ar), 125.37 (Ar), 123.34 (Ar), 117.86 (Ar), 34.32 (Ar*C*(CH$_3$)$_3$), 31.58 (ArC(*C*H$_3$)$_3$), 28.21 (Ar*C*H(CH$_3$)$_2$), 23.67 (ArCH(*C*H$_3$)$_2$), 23.63 (ArCH(*C*H$_3$)$_2$), 20.91 (Ar*C*H$_3$), 18.55 (Ar*C*H$_3$) ppm. HRMS (ES+) Calcd. for $C_{55}H_{71}N_2O_2$: 791.5516. Found: 791.5518.

10-s. The imine condensation with 2,6-diisopropylaniline and **9-s** was accomplished via the same procedure as for the anti. The desired product was isolated as a pale yellow solid in 42 % yield (0.17 g). ^{1}H NMR (500 MHz, $CDCl_3$): δ 12.93 (s, 2H, ArO*H*), 8.36 (s, 2H, C*H*N), 7.42 (d, J = 2.5 Hz, 2H, Ar*H*), 7.31 (d, J = 2.5 Hz, 2H, Ar*H*), 7.18 (m, 7H, Ar*H*), 3.04 (hept, J = 6.9 Hz, 4H, ArC*H*(CH$_3$)$_2$), 2.19 (s, 6H, ArCH_3), 1.99 (s, 3H, ArCH_3), 1.39 (s, 18H, ArC(CH_3)$_3$), 1.19 (d, J = 6.9 Hz, 12H, ArCH(CH_3)$_2$), 1.17 (d, J = 6.9 Hz, 12H, ArCH(CH_3)$_2$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 166.99 (N*C*H), 156.61 (Ar), 146.61 (Ar), 141.37 (Ar), 138.91 (Ar), 136.25 (Ar), 136.09 (Ar), 135.41 (Ar), 132.87 (Ar), 129.43 (Ar), 129.03 (Ar), 127.38 (Ar), 125.30 (Ar), 123.30 (Ar), 117.98 (Ar), 34.31 (Ar*C*(CH$_3$)$_3$), 31.62 (ArC(*C*H$_3$)$_3$), 28.17 (Ar*C*H(CH$_3$)$_2$), 23.76 (ArCH(*C*H$_3$)$_2$), 23.63 (ArCH(*C*H$_3$)$_2$), 20.93 (Ar*C*H$_3$), 18.60 (Ar*C*H$_3$) ppm. HRMS (FAB+) Calcd. for $C_{55}H_{71}N_2O_2$: 791.5516. Found: 791.5515.

10-s-OMe. The imine condensation with 2,6-diisopropylaniline and **9-s-OMe** was accomplished via the same procedure as for **6-a**. The desired product was isolated as a pale yellow solid in 47 % yield (2.39 g). ^{1}H NMR (400 MHz, C_6D_6): δ 13.35 (s, 2H, ArO*H*), 8.04 (s,

2H, NC*H*) 7.49 (d, J = 1.6 Hz, 2H, Ar*H*), 7.26 (d, J = 1.6 Hz, 2H, Ar*H*), 7.10 (s, 6H, Ar*H*), 3.44 (s, 3H, ArOCH_3), 3.02 (septet, J = 6.8 Hz, 4H, ArC*H*(CH_3)$_2$), 2.42 (s, 6H, ArCH_3), 2.37 (s, 3H, ArCH_3), 1.22 (s, 18H, ArC(CH_3)$_3$), 1.05 (d, J = 7.0 Hz, 12H, ArCH(CH_3)$_2$), 1.03 (d, J = 7.0 Hz, 12H, ArCH(CH_3)$_2$) ppm. ^{13}C NMR (101 MHz, C_6D_6): δ 167.81 (Ar), 157.57 (Ar), 155.81 (Ar), 147.28 (Ar), 141.69 (Ar), 138.95 (Ar), 137.37 (Ar), 133.37 (Ar), 131.45 (Ar), 130.71 (Ar), 129.47 (Ar), 127.83 (Ar), 125.74 (Ar), 123.48 (Ar), 118.74 (Ar), 59.50 (O*C*H$_3$), 34.16 (Ar*C*(CH$_3$)$_3$), 31.54 (ArC(*C*H$_3$)$_3$), 28.58 (Ar*C*H(CH$_3$)$_2$), 23.53 (ArCH(*C*H$_3$)$_2$), 23.44 (ArCH(*C*H$_3$)$_2$), 18.98 (Ar*C*H$_3$), 14.52 (Ar*C*H$_3$) ppm. HRMS (ES+) Calcd. for $C_{56}H_{73}N_2O_3$: 821.5621. Found: 821.5641.

Synthesis of dinickel complexes



Scheme S3: Synthesis of dinickel complexes

Ni(PMe$_3$)$_2$MeCl[9,10] was synthesized according to literature procedures.

1-a. Prior to metallation, **6-a** was deprotonated with Na(N(SiMe$_3$)$_2$). A scintillation vial equipped with a stir bar was charged with Na(N(SiMe$_3$)$_2$) (0.018 g, 0.099 mmol, 2 equiv) and toluene (1 mL). A solution of **6-a** (0.040 g, 0.050 mmol) in toluene (2 mL) was added and the mixture was stirred at room temperature for 2 h, and then concentrated *in vacuo* to yield a bright yellow solid. The amine side product was removed by two cycles of suspending the product in hexanes and removing the volatiles under vacuum. The solid was used without further purification. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37 (s, 2H, NC*H*), 7.39 (d, *J*=2.5 Hz, 2H, Ar*H*), 7.31 (d, *J*=2.5 Hz, 2H, Ar*H*), 7.18 (m, 6H, Ar*H*), 3.06 (hept, *J*=6.7 Hz, 4H, C*H*(CH$_3$)$_2$), 2.09 (s, 12H ArCH_3), 1.35 (s, 18H, C(CH_3)$_3$), 1.19 (d, *J*=6.7 Hz, 24H, CH(CH_3)$_2$) ppm.

Metallations with Ni(PMe$_3$)$_2$MeCl were carried out using literature conditions.[11] A solution of deprotonated **6-a** (0.114 g, 0.134 mmol, 1 equiv) in Et_2O (4 mL) was added dropwise to a solution of Ni(PMe$_3$)$_2$MeCl (0.070 g, 0.266 mmol, 1.98 equiv) in Et_2O (3 mL) and the resulting solution was stirred for 14 h, and then concentrated *in vacuo* to yield a dark orange brown solid. The solid was suspended in hexanes and filtered over Celite. The hexanes washes were

discarded and the desired product was flushed through the Celite with benzene and then concentrated *in vacuo* to yield 0.12 g of bright orange solid (81 % yield). ^{1}H NMR (600 MHz, C_6D_6): δ 8.02 (d, *J*=8.9 Hz, 2H, NC*H*), 7.38 (d, *J*=2.7 Hz, 2H, Ar*H*), 7.15 (m, 8H, Ar*H*), 3.93 (hept, *J*=6.9 Hz, 4H, C*H*(CH_3)$_2$), 2.15 (s, 12H, ArCH_3), 1.38 (s, 18H, C(CH_3)$_3$), 1.37 (d, *J*=6.9 Hz, 12H, CH(CH_3)$_2$), 1.05 (d, *J*=6.9 Hz, 12H, CH(CH_3)$_2$), 0.70 (d, *J*=9.7 Hz, 18H, P(CH_3)$_3$), -1.14 (d, *J*=7.2 Hz, 6H, NiCH_3) ppm. ^{31}P NMR (121 MHz, C_6D_6): δ -8.36 (s) ppm. ^{13}C NMR (126 MHz, C_6D_6): δ 165.88 (N*C*H), 163.83 (Ar), 150.03 (Ar), 141.47 (Ar), 139.97 (Ar), 136.02 (Ar), 135.47 (Ar), 132.99 (Ar), 132.36 (Ar), 128.35 (Ar), 126.29 (Ar), 123.60 (Ar), 118.76 (Ar), 33.90 (Ar*C*(CH_3)$_3$), 31.76 (ArC(*C*H_3)$_3$), 28.55 (Ar*C*H(CH_3)$_2$), 25.00 (ArCH(*C*H_3)$_2$), 23.22 (ArCH(*C*H_3)$_2$), 18.28 (Ar*C*H_3), 12.48 (d, *J*=27.38 Hz, P(*C*H_3)$_3$), -14.13 (d, *J*=42.36 Hz, Ni*C*H_3) ppm. Anal. Calcd for $C_{64}H_{94}N_2Ni_2O_2P_2$: C, 69.70; H, 8.59, N, 2.54. Found: C, 69.56; H, 8.50, N, 2.57.

1-s. Prior to metallation, **6-s** was deprotonated with KH. A scintillation vial equipped with a stir bar was charged with KH (0.005 g, 0.117 mmol, 2 equiv) and THF (1 mL) and frozen in the glovebox cold well. A solution of **6-s** (0.050 g, 0.059 mmol) in THF (2 mL) was also frozen in the cold well. The solution of **6-s** was added and the solution of KH while thawing, and the mixture was stirred at room temperature for 2 h, and then concentrated *in vacuo* to yield a bright yellow solid. The solid was taken up in Et_2O, filtered over Celite, concentrated *in vacuo*, and used without further purification. ^{1}H NMR (300 MHz, C_6D_6): δ 8.17 (s, 2H, NC*H*), 7.36 (d, *J*=2.7 Hz, 2H, Ar*H*), 7.29 (d, *J*=2.7 Hz, 2H, Ar*H*), 7.07 (bs, 6H, Ar*H*), 3.05 (hept, *J*=6.8 Hz, 4H, C*H*(CH_3)$_2$), 2.06 (s, 12H, ArCH_3), 1.32 (s, 18H, C(CH_3)$_3$), 1.09 (d, *J*=6.8 Hz, 24H, CH(CH_3)$_2$) ppm.

A solution of deprotonated **6-s** (0.151 g, 0.171 mmol, 1 equiv) in Et_2O (5 mL) was added dropwise to a solution of Ni(PMe_3)$_2$MeCl (0.089 g, 0.340 mmol, 1.98 equiv) in Et_2O (4 mL) and the resulting solution was stirred for 14 h, filtered over Celite, and then concentrated *in vacuo* to yield a red orange solid. The ^{1}H and ^{31}P NMR spectra still indicated the presence of Ni(PMe_3)$_2$MeCl so an additional 0.15 equiv of deprotonated **6-s** was added to the red orange solid in Et_2O (9 mL) and the reaction was stirred an additional 14 h, filtered over Celite, and then concentrated *in vacuo*. 0.16 g of red orange solid was collected (84 % yield). ^{1}H NMR (600 MHz, C_6D_6): δ 8.04 (s, 2H, NC*H*), 7.34 (d, *J*=2.8 Hz, 2H, Ar*H*), 7.15 (bs, 6H, Ar*H*), 7.10 (d, *J*=2.8 Hz, 2H, Ar*H*), 3.95 (hept, *J*=6.9 Hz, 4H, C*H*(CH_3)$_2$), 2.24 (s, 12H, ArCH_3), 1.34 (d, *J*=6.9 Hz, 12H, CH(CH_3)$_2$), 1.24 (s, 18H, C(CH_3)$_3$), 1.03 (d, *J*=6.9 Hz, 12H, CH(CH_3)$_2$), 0.80 (d, *J*=9.5 Hz, 18H, P(CH_3)$_3$), -1.06 (s, 6H, NiCH_3) ppm. ^{31}P NMR (121 MHz, C_6D_6): δ -7.92 (s) ppm. ^{13}C NMR (126 MHz, C_6D_6): δ 166.48 (N*C*H), 164.19 (Ar), 149.84 (Ar), 141.61 (Ar), 140.53 (Ar), 135.85 (Ar), 135.82 (Ar), 135.44 (Ar), 132.85 (Ar), 128.96 (Ar), 126.34 (Ar), 123.66 (Ar), 119.09 (Ar), 33.84 (Ar*C*(CH_3)$_3$), 31.57 (ArC(*C*H_3)$_3$), 28.53 (Ar*C*H(CH_3)$_2$), 25.08 (ArCH(*C*H_3)$_2$), 23.18 (ArCH(*C*H_3)$_2$), 19.08 (Ar*C*H_3), 13.17 (d, *J*=27.82 Hz, P(*C*H_3)$_3$), -12.70 (d, *J*=44.90 Hz, Ni*C*H_3) ppm. Anal. Calcd for $C_{64}H_{94}N_2Ni_2O_2P_2$: C, 69.70; H, 8.59, N, 2.54. Found: C, 69.64; H, 8.62, N, 2.51.

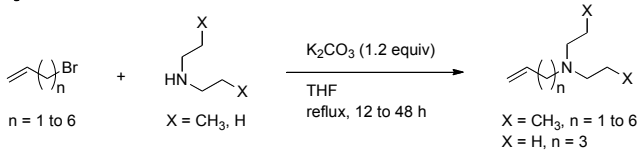
2-a. Deprotonation of **10-a** was accomplished via the same procedure as was used to deprotonate **6-s** (with KH). The deprotonated material was then dissolved in Et_2O, filtered over Celite, and metallated with Ni(PMe_3)$_2$MeCl using the same procedure as for **1-a**. **2-a** was purified by precipitation from pentane. ^{1}H NMR (500 MHz, C_6D_6): δ 7.99 (d, *J*=8.6 Hz, 2H, NC*H*), 7.34 (d, *J*=2.7 Hz, 2H, Ar*H*), 7.14 (m, 6H, Ar*H*), 7.10 (d, *J*=2.7 Hz, 2H, Ar*H*), 7.03 (s, 1H, ArH), 3.90 (ddt, *J*=13.8, 9.7, 6.8 Hz, 4H, C*H*(CH_3)$_2$), 2.26 (s, 6H, ArCH_3), 2.14 (s, 3H, ArCH_3), 1.37 (d, *J*=6.9 Hz, 6H, CH(CH_3)$_2$), 1.35 (d, *J*=6.9 Hz, 6H, CH(CH_3)$_2$), 1.34 (s, 18H, C(CH_3)$_3$), 1.05 (d, *J*=6.9 Hz, 6H, CH(CH_3)$_2$), 1.01 (d, *J*=6.9 Hz, 6H, CH(CH_3)$_2$), 0.77 (d, *J*=9.7

Hz, 18H, P(CH_3)$_3$), -1.14 (d, *J*=6.9 Hz, 6H, NiCH_3) ppm. ^{31}P NMR (121 MHz, C_6D_6): δ -7.81 (s) ppm. ^{13}C NMR (126 MHz, C_6D_6): δ 165.45 (N*C*H), 163.33 (Ar), 149.61 (Ar), 141.11 (Ar), 141.02 (Ar), 138.43 (Ar), 135.90 (Ar), 135.04 (Ar), 134.44 (Ar), 134.39 (Ar) 132.58 (Ar), 128.09 (Ar), 125.87 (Ar), 123.16 (Ar), 118.41 (Ar), 33.49 (Ar*C*(CH$_3$)$_3$), 31.33 (ArC(*C*H$_3$)$_3$), 28.14 (Ar*C*H(CH$_3$)$_2$), 24.60 (ArCH(*C*H$_3$)$_2$), 24.50 (ArCH(*C*H$_3$)$_2$), 22.82 (ArCH(*C*H$_3$)$_2$), 22.74 (ArCH(*C*H$_3$)$_2$), 20.88 (Ar*C*H$_3$), 18.08 (Ar*C*H$_3$), 12.25 (d, *J*=27.39 Hz, P(*C*H$_3$)$_3$), -14.54 (d, *J*=42.19 Hz, Ni*C*H$_3$) ppm. Anal. Calcd for $C_{63}H_{92}N_2Ni_2O_2P_2$: C, 69.50; H, 8.52, N, 2.57. Found: C, 69.45; H, 8.56, N, 2.59.

2-s. Deprotonation of **10-s** was accomplished via the same procedure as was used to deprotonate **6-s** (with KH). The deprotonated material was then dissolved in hexanes, filtered over Celite, and metallated with Ni(PMe$_3$)$_2$MeCl using the same procedure as for **1-s**. **2-s** was purified by recrystallization from pentane. ^{1}H NMR (500 MHz, C_6D_6): δ 8.01 (d, *J*=8.4 Hz, 2H, NC*H*), 7.42 (d, *J*=2.8 Hz, 2H, Ar*H*), 7.17 (m, 6H, Ar*H*), 7.07 (d, *J*=2.8 Hz, 2H, Ar*H*), 7.02 (s, 1H, Ar*H*), 3.97 (hept, *J*=6.8 Hz, 4H, C*H*(CH$_3$)$_2$), 2.35 (s, 6H, ArC*H*$_3$), 2.25 (s, 3H, ArC*H*$_3$), 1.43 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.40 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.22 (s, 18H, C(C*H*$_3$)$_3$), 1.07 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.02 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 0.92 (d, *J*=9.6 Hz, 18H, P(C*H*$_3$)$_3$), -1.06 (d, *J*=6.6 Hz, 6H, NiC*H*$_3$) ppm. ^{31}P NMR (121 MHz, C_6D_6): δ -8.58 (s) ppm. ^{13}C NMR (126 MHz, C_6D_6): δ 165.99 (N*C*H), 163.42 (Ar), 149.45 (Ar), 141.20 (Ar), 141.09 (Ar), 139.27 (Ar), 135.99 (Ar), 135.47 (Ar), 135.36 (Ar), 133.85 (Ar) 128.73 (Ar), 125.94 (Ar), 123.33 (Ar), 123.19 (Ar), 118.89 (Ar), 33.38 (Ar*C*(CH$_3$)$_3$), 31.11 (ArC(*C*H$_3$)$_3$), 28.18 (Ar*C*H(CH$_3$)$_2$), 24.72 (ArCH(*C*H$_3$)$_2$), 24.58 (ArCH(*C*H$_3$)$_2$), 22.92 (ArCH(*C*H$_3$)$_2$), 22.87 (ArCH(*C*H$_3$)$_2$), 21.34 (Ar*C*H$_3$), 20.34 (Ar*C*H$_3$), 13.22 (d, *J*=27.75 Hz, P(*C*H$_3$)$_3$), -12.97 (d, *J*=42.27 Hz, Ni*C*H$_3$) ppm. Anal. Calcd for $C_{63}H_{92}N_2Ni_2O_2P_2$: C, 69.50; H, 8.52, N, 2.57. Found: C, 69.45; H, 8.43, N, 2.53.

2-s-OMe. Deprotonation of **10-s-OMe** was accomplished via the same procedure as was used to deprotonate **6-s** (with KH). The deprotonated material was then dissolved in hexanes, filtered over Celite, and metallated with Ni(PMe$_3$)$_2$MeCl using the same procedure as for **1-s**. **2-s-OMe** was purified by precipitation from cold hexanes. The assignment of this product as the syn atropisomer was confirmed by ^{1}H-^{1}H NOESY NMR. ^{1}H NMR (600 MHz, C_6D_6): δ 8.01 (d, *J*=7.8 Hz, 2H, NC*H*), 7.42 (d, *J*=2.8 Hz, 2H, Ar*H*), 7.17 (m, 6H, Ar*H*), 7.09 (d, *J*=2.8 Hz, 2H, Ar*H*), 3.97 (hept, *J*=6.8 Hz, 4H, C*H*(CH$_3$)$_2$), 3.54 (s, 3H, ArOC*H*$_3$), 2.40 (s, 6H, ArC*H*$_3$), 2.19 (s, 3H, ArC*H*$_3$), 1.43 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.39 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.23 (s, 18H, C(C*H*$_3$)$_3$), 1.08 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 1.02 (d, *J*=6.9 Hz, 6H, CH(C*H*$_3$)$_2$), 0.93 (d, *J*=9.5 Hz, 18H, P(C*H*$_3$)$_3$), -1.07 (d, *J*=5.7 Hz, 6H, NiC*H*$_3$) ppm. ^{31}P NMR (121 MHz, C_6D_6): δ -8.66 (s) ppm. ^{13}C NMR (126 MHz, C_6D_6): δ 165.98 (N*C*H), 163.38 (Ar), 154.70 (Ar), 149.43 (Ar), 141.20 (Ar), 141.06 (Ar), 140.70 (Ar), 135.39 (Ar), 133.89 (Ar), 131.65 (Ar), 128.80 (Ar), 125.95 (Ar), 123.35 (Ar), 123.17 (Ar), 118.94 (Ar), 58.99 (ArO*C*H$_3$), 33.38 (Ar*C*(CH$_3$)$_3$), 31.12 (ArC(*C*H$_3$)$_3$), 28.19 (Ar*C*H(CH$_3$)$_2$), 28.16 (Ar*C*H(CH$_3$)$_2$), 24.73 (ArCH(*C*H$_3$)$_2$), 24.58 (ArCH(*C*H$_3$)$_2$), 22.92 (ArCH(*C*H$_3$)$_2$), 22.86 (ArCH(*C*H$_3$)$_2$), 20.26 (Ar*C*H$_3$), 14.65 (Ar*C*H$_3$), 13.18 (d, *J*=27.73 Hz, P(*C*H$_3$)$_3$), -13.03 (d, *J*=44.35 Hz, Ni*C*H$_3$) ppm. Anal. Calcd for $C_{64}H_{94}N_2Ni_2O_3P_2$: C, 68.71; H, 8.47, N, 2.50. Found: C, 68.66; H, 8.39, N, 2.47.

Synthesis of amino olefins.



Scheme S4. General synthesis of amino olefins.

Representative procedure: synthesis of N(pentenyl)(^{n}Pr)$_2$. Under ambient conditions, K_2CO_3 (7.00 g, 50.65 mmol, 1.2 equiv), THF (42 mL), HN(^{n}Pr)$_2$ (7.52 mL, 54.87 mmol, 1.3 equiv), and 5-bromo-1-pentene (5.00 mL, 42.21 mmol, 1 equiv) were added, in that order, to a Schlenk tube equipped with a stirbar. The reaction vessel was sealed and heated to 75 °C for 40 h. The complete consumption of the bromide was confirmed by GCMS. The reaction mixture was filtered and the salts were rinsed with THF. The filtrates were then combined and fractionally distilled under static vacuum to separate the product from solvent, excess starting amine, and non-volatile side products. The clear, colorless oil collected in the second fraction was filtered over about 30 mL of silica gel in a frit with about 150 mL of pentane. This pentane fraction was then fractionally distilled under static vacuum to separate the product from pentane. The product was obtained as a clear, colorless oil in the second fraction and was dried by stirring over CaH_2 overnight, degassing on the Schlenk line, bringing into the glovebox and filtering over an alumina pipette plug (3.45 g, 48 % yield).

N(allyl)(^{n}Pr)$_2$. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.84 (m, 1H, C*H*), 5.11 (m, 2H, CH_2), 3.06 (dt, *J*=6.5 Hz, 1.4, 2H, NCH_2), 2.36 (m, 4H, NCH_2), 1.43 (m, 4H, CH_2), 0.85 (t, *J*=7.4 Hz, 6H, CH_3) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.50 (olefinic), 116.78 (olefinic), 57.54 (N*C*H$_2$), 56.01 (N*C*H$_2$), 20.35 (*C*H$_2$), 12.05 (*C*H$_3$) ppm. HRMS (FAB) Calcd. for $C_9H_{20}N$: 142.1596. Found: 142.1597.

N(butenyl)(^{n}Pr)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.80 (m, 1H, C*H*), 5.00 (m, 2H, CH_2), 2.49 (m, 2H, NCH_2), 2.38 (m, 4H, NCH_2), 2.19 (m, 2H, CH_2), 1.44 (m, 4H, CH_2), 0.87 (t, *J*=7.4 Hz, 6H, CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 137.37 (olefinic), 115.37 (olefinic), 56.30 (N*C*H$_2$), 53.75 (N*C*H$_2$), 31.65 (*C*H$_2$), 20.43 (*C*H$_2$), 12.14 (*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{10}H_{21}N$: 155.1674. Found: 155.1690.

N(pentenyl)(^{n}Pr)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.82 (m, 1H, C*H*), 4.96 (m, 2H, CH_2), 2.40 (m, 2H, NCH_2), 2.35 (m, 4H, NCH_2), 2.04 (m, 2H, CH_2), 1.52 (m, 2H, CH_2), 1.43 (m, 4H, CH_2), 0.86 (t, *J*=7.3 Hz, 6H, CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 139.01 (olefinic), 114.47 (olefinic), 56.45 (N*C*H$_2$), 53.83 (N*C*H$_2$), 31.90 (*C*H$_2$), 26.51 (*C*H$_2$), 20.42 (*C*H$_2$), 12.13 (*C*H$_3$) ppm. HRMS (EI+) Calcd. for $C_{11}H_{23}N$: 169.1830. Found: 169.1837.

N(hexenyl)(^{n}Pr)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.81 (m, 1H, C*H*), 4.97 (m, 2H, CH_2), 2.39 (m, 2H, NCH_2), 2.35 (m, 4H, NCH_2), 2.06 (m, 2H, CH_2), 1.41 (m, 8H, CH_2), 0.86 (t, *J*=7.4 Hz, 6H, CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 139.16 (olefinic), 114.43 (olefinic), 56.45 (N*C*H$_2$), 54.21 (N*C*H$_2$), 33.90 (*C*H$_2$), 27.09 (*C*H$_2$), 26.72 (*C*H$_2$), 20.41 (*C*H$_2$), 12.16 (*C*H$_3$) ppm. HRMS (FAB) Calcd. for $C_{12}H_{26}N$: 184.2065. Found: 184.2056.

N(heptenyl)(^{n}Pr)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.80 (m, 1H, C*H*), 4.96 (m, 2H, CH_2), 2.36 (m, 6H, NCH_2), 2.04 (m, 2H, CH_2), 1.42 (m, 8H, CH_2), 1.28 (m, 2H, CH_2), 0.86 (t, *J*=7.4 Hz, 6H, CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 139.25 (olefinic), 114.33 (olefinic), 56.47 (N*C*H$_2$), 54.39 (N*C*H$_2$), 33.96 (*C*H$_2$), 29.08 (*C*H$_2$), 27.30 (*C*H$_2$), 27.11 (*C*H$_2$), 20.39 (*C*H$_2$), 12.16 (*C*H$_3$) ppm. HRMS (FAB) Calcd. for $C_{13}H_{28}N$: 198.2222. Found: 198.2227.

N(octenyl)(^{n}Pr)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.80 (m, 1H, C*H*), 4.95 (m, 2H, CH_2), 2.36 (m, 6H, NCH_2), 2.03 (m, 2H, CH_2), 1.39 (m, 14H, CH_2), 0.86 (t, *J*=7.4 Hz, 6H, CH_3) ppm.

^{13}C NMR (126 MHz, $CDCl_3$): δ 139.32 (olefinic), 114.27 (olefinic), 56.46 (N*C*H_2), 54.44 (N*C*H_2), 33.92 (*C*H_2), 29.28 (*C*H_2), 29.08 (*C*H_2), 27.66 (*C*H_2), 27.21 (*C*H_2), 20.39 (*C*H_2), 12.16 (*C*H_3) ppm. HRMS (FAB) Calcd. for $C_{14}H_{30}N$: 212.2378. Found: 212.2381.

N(pentenyl)(Et)$_2$. ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.81 (m, 1H, C*H*), 4.97 (m, 2H, C*H*$_2$), 2.50 (q, *J*=7.2 Hz, 4H, NC*H*$_2$), 2.40 (m, 2H, NC*H*$_2$), 2.03 (m, 2H, C*H*$_2$), 1.53 (m, 2H, C*H*$_2$), 1.00 (t, *J*=7.2 Hz, 6H, C*H*$_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$): δ 138.84 (olefinic), 114.55 (olefinic), 52.53 (N*C*H_2), 47.06 (N*C*H_2), 31.95 (*C*H_2), 26.36 (*C*H_2), 11.86 (*C*H_3) ppm. HRMS (FAB) Calcd. for $C_9H_{20}N$: 142.1596. Found: 142.1581.

General polymerization procedures. A 3 oz. Andrews glass pressure reaction vessel equipped with Swagelock valves and a gauge was used for all high pressure polymerizations. A syringe was loaded with a solution of the desired organometallic complex and scavenger, and the needle was sealed with a rubber septum. The high-pressure setup was brought into the glove box with a magnetic stirbar and charged with the desired amount of solvent (minus that which was used to make the solution of complex and scavenger). The desired amount of additive or comonomer was also added to the setup, if applicable, and the setup was sealed. The syringe and setup were brought out of the box and the setup was clamped firmly over a hot plate with a mineral oil bath previously regulated to the desired temperature (25 °C). The solution was stirred vigorously (1200 rpm). A nylon core hose equipped with quick connect adaptors was purged with ethylene for 1 minute and the pressure was set to 15 psig. The hose was connected to the setup and the setup was filled with ethylene. A bleed needle was inserted into a Teflon septum at the top of the high pressure setup and flushed with ethylene. The solution of organometallic complex and scavenger was added via syringe and the top of the setup was closed. The pressure was increased to the desired level (100 psig). After the desired time, the ethylene hose was disconnected, the setup was vented and the reaction mixture was quenched with acidified methanol (3 times the reaction volume) to precipitate the polymer, which was collected as a white solid by filtration over a fine frit. If only a small amount of polymer was precipitated, the entire mixture was collected and volatile materials were removed under vacuum. All polymers were dried on the Schlenk line for a minimum of 8 hours before a mass was recorded.

NMR Data

^{1}H and ^{13}C NMR spectra of synthesized compounds

Figure S1. ^{1}H NMR spectrum of **3-a** and **3-s** in $CDCl_3$.

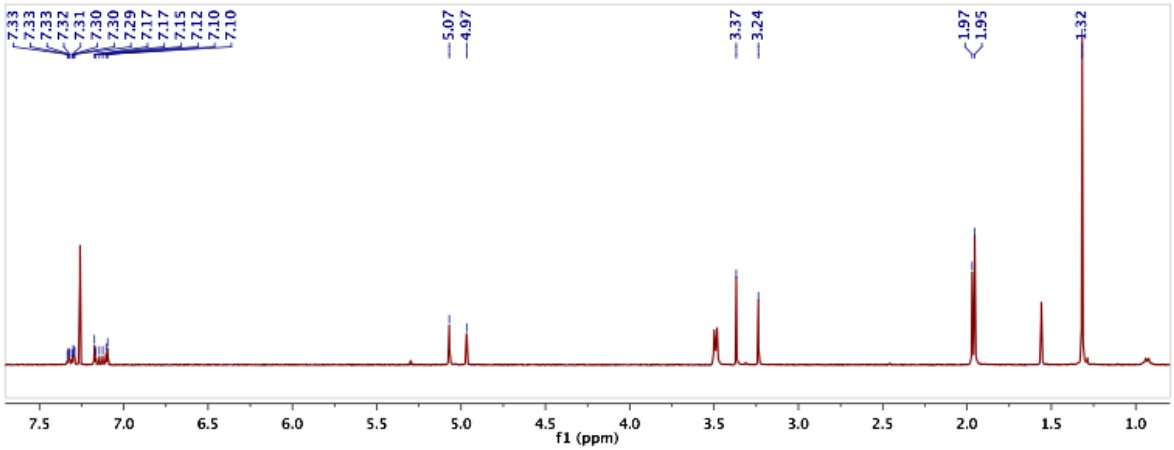


Figure S2. ^{1}H NMR spectrum of **4-a** and **4-s** in $CDCl_3$ (impurities include TMEDA).

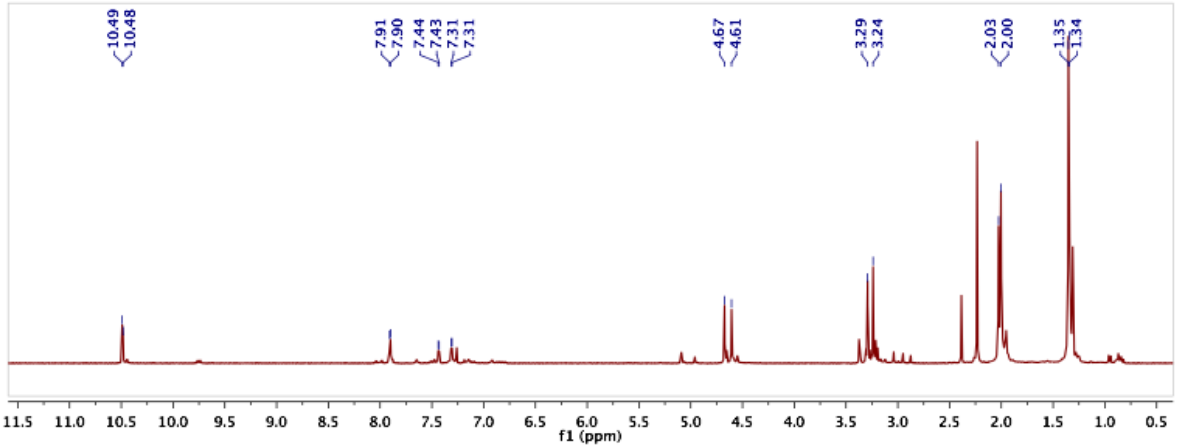


Figure S3. ^{1}H NMR spectrum of 3,5-dibromo-2,4,6-trimethyl-methoxybenzene in $CDCl_3$.

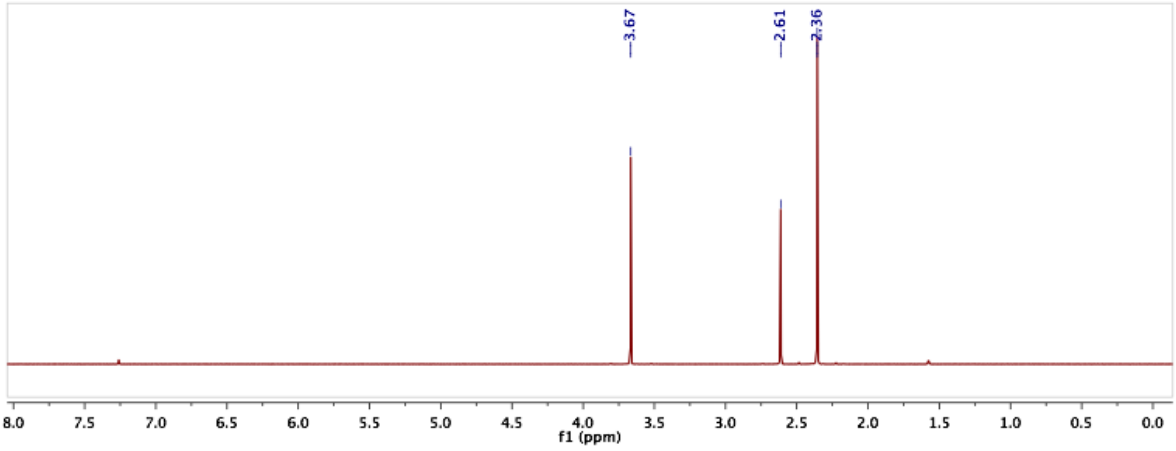


Figure S4. ^{13}C NMR spectrum of 3,5-dibromo-2,4,6-trimethyl-methoxybenzene in $CDCl_3$.

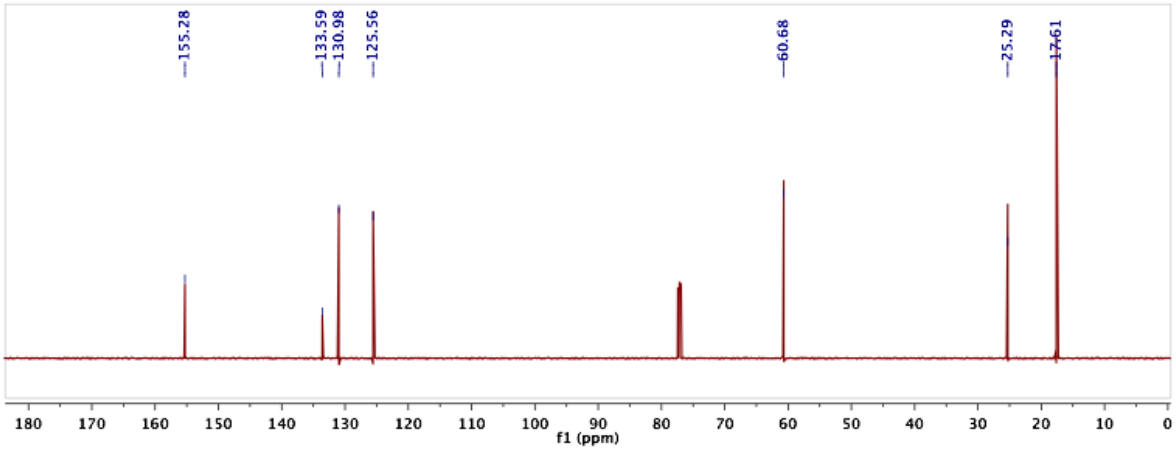


Figure S5. ^{1}H NMR spectrum of **7-a** in $CDCl_3$.

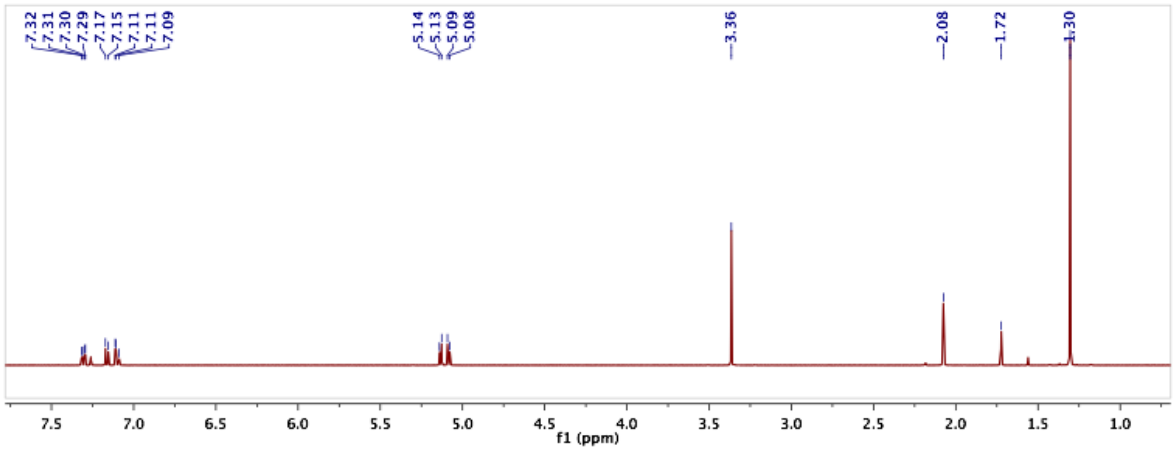


Figure S6. ^{13}C NMR spectrum of **7-a** in $CDCl_3$.

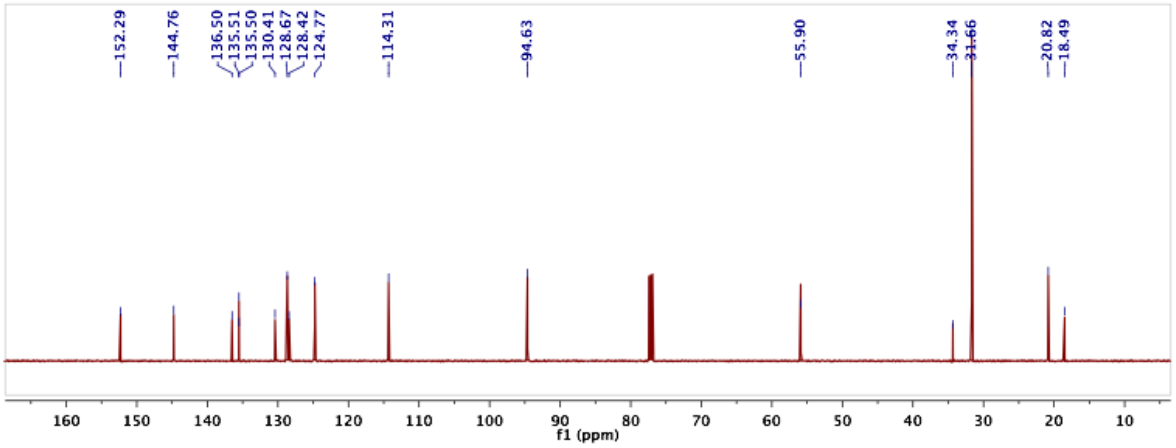


Figure S7. ^{1}H NMR spectrum of **7-s** in $CDCl_3$.

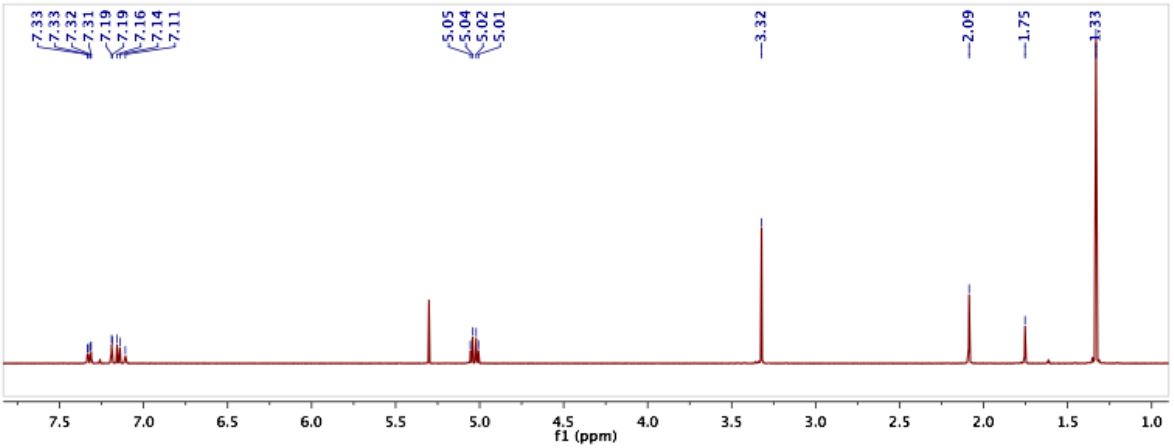


Figure S8. ^{13}C NMR spectrum of **7-s** in $CDCl_3$.

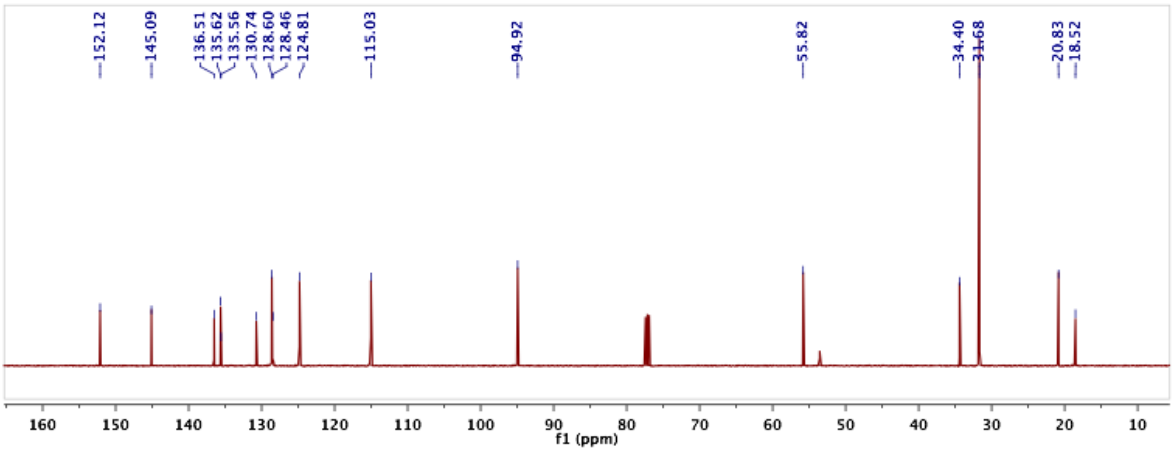


Figure S9. ^{1}H NMR spectrum of **7-s-OMe** in C_6D_6.

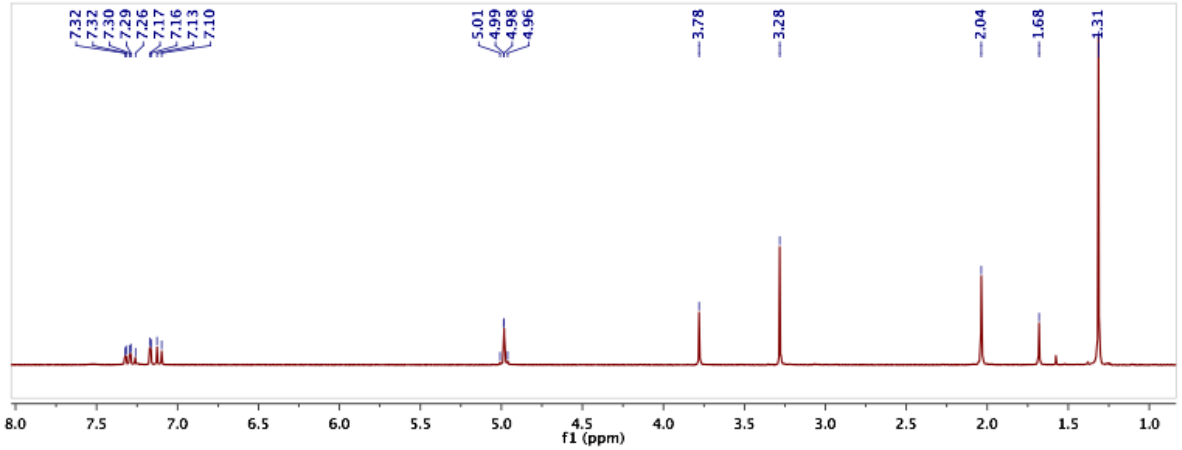


Figure S10. ^{13}C NMR spectrum of **7-s-OMe** in C_6D_6.

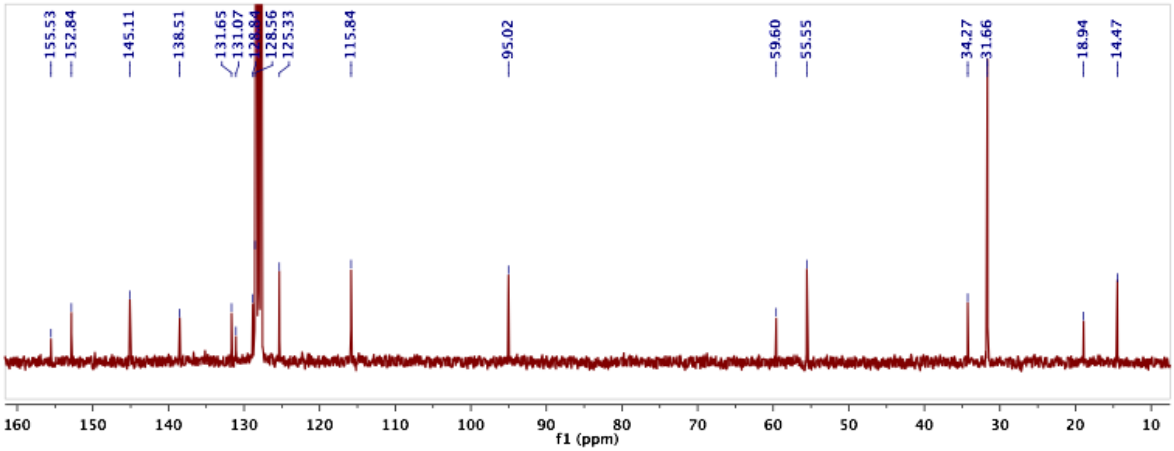


Figure S11. ^{1}H NMR spectrum of **8-a** in $CDCl_3$.

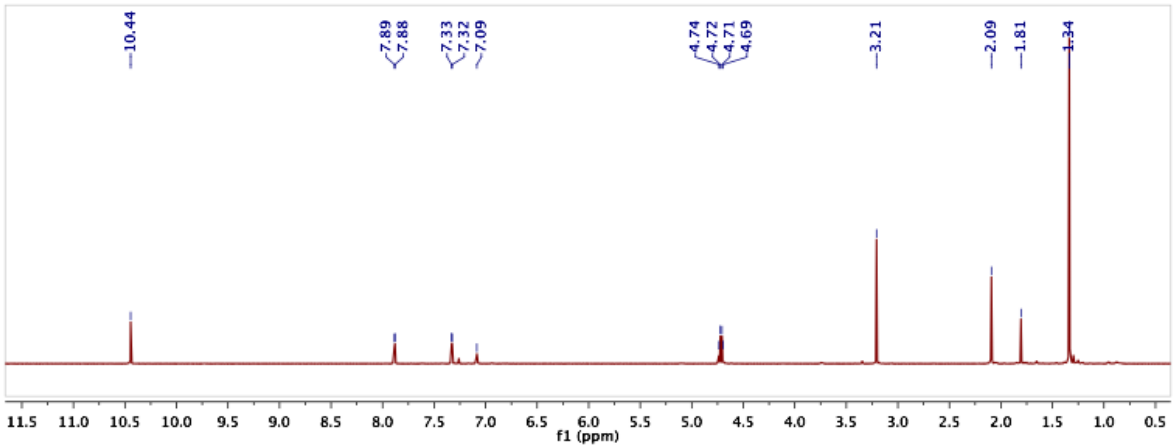


Figure S12. ^{13}C NMR spectrum of **8-a** in $CDCl_3$.

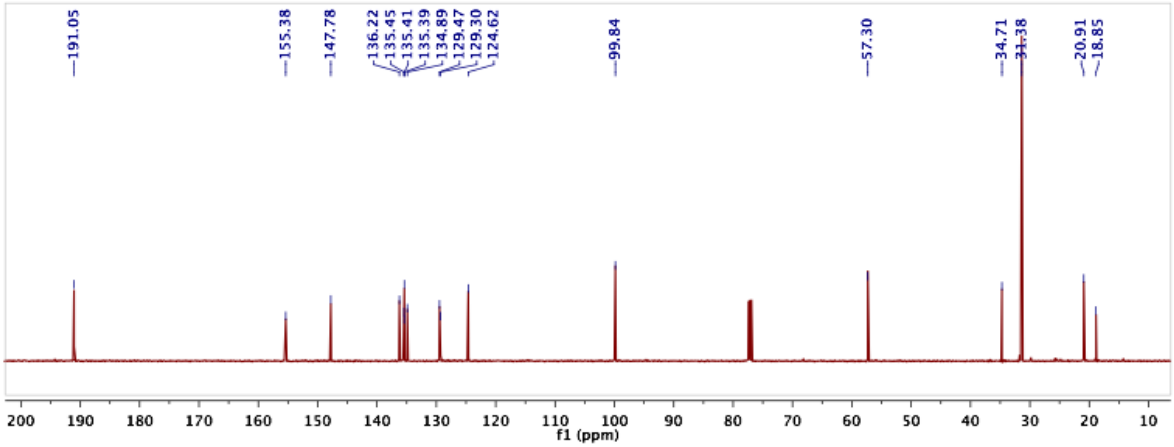


Figure S13. ^{1}H NMR spectrum of **8-s** in $CDCl_3$.

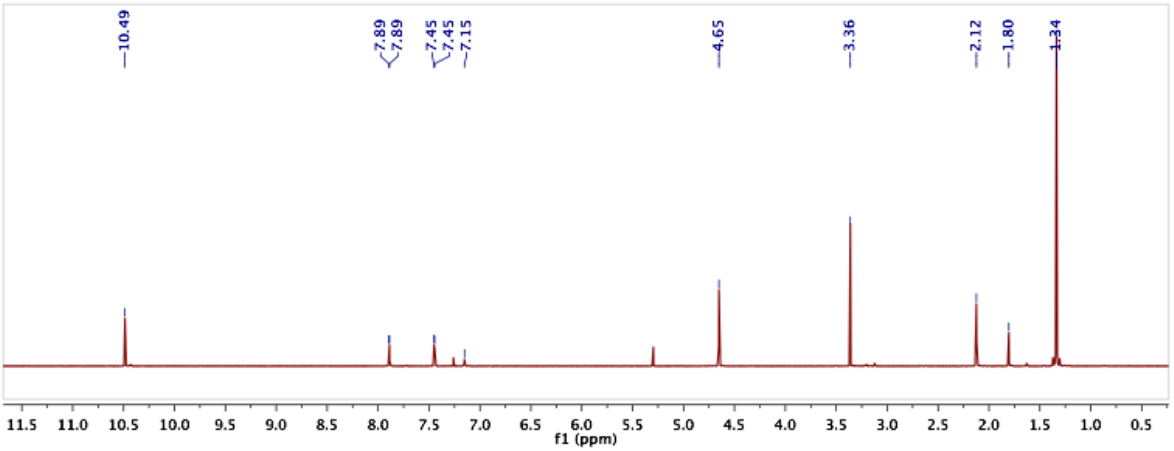


Figure S14. ^{13}C NMR spectrum of **8-s** in $CDCl_3$.

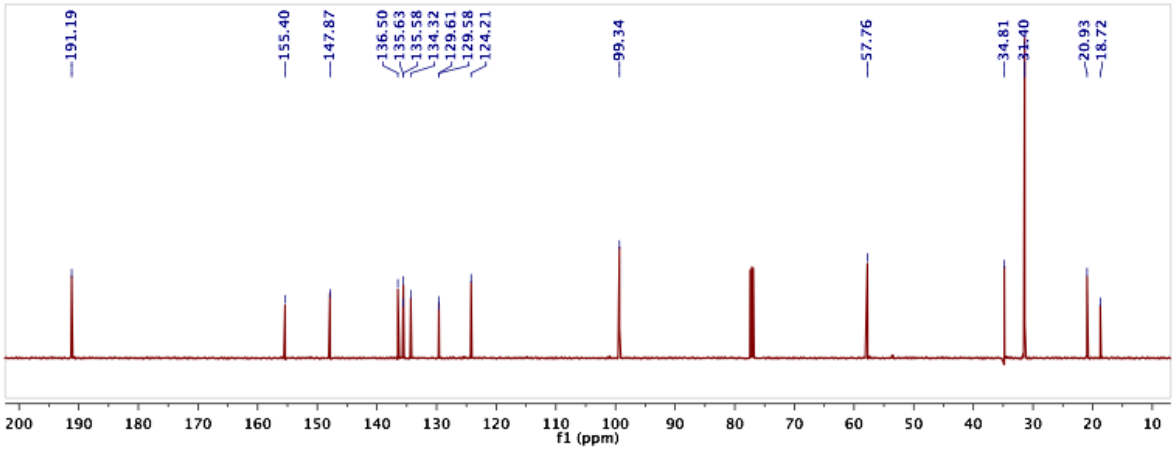


Figure S15. ^{1}H NMR spectrum of **8-s-OMe** in $CDCl_3$.

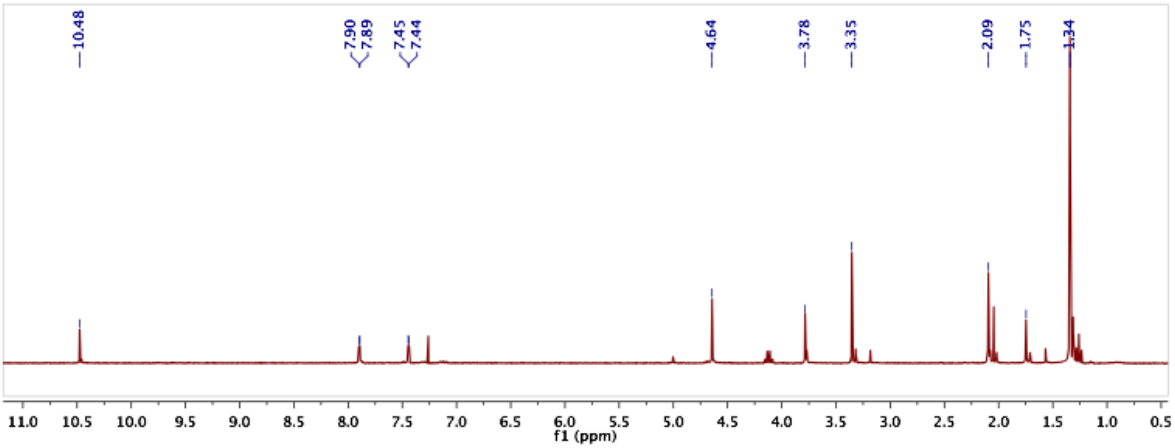


Figure S16. ^{1}H NMR spectrum of **9-a** in $CDCl_3$.

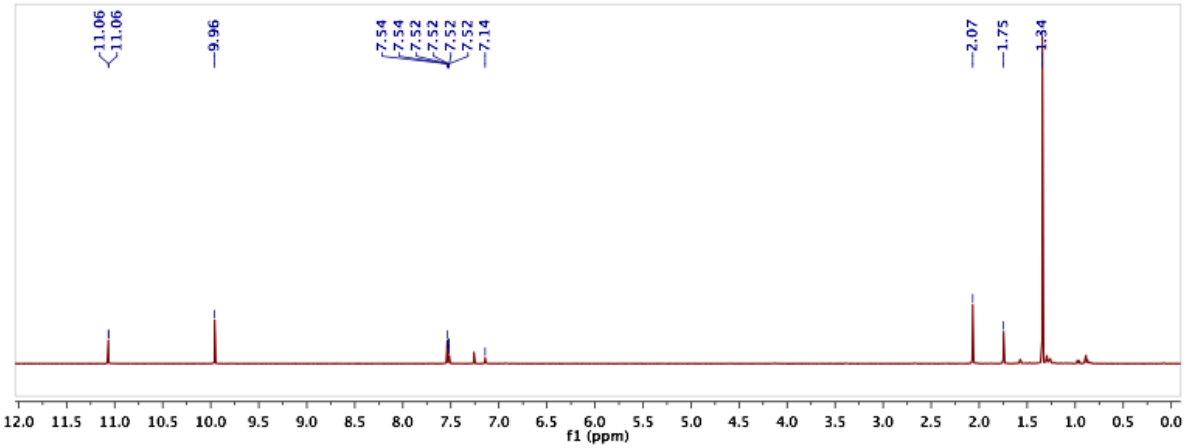


Figure S17. ^{13}C NMR spectrum of **9-a** in $CDCl_3$.

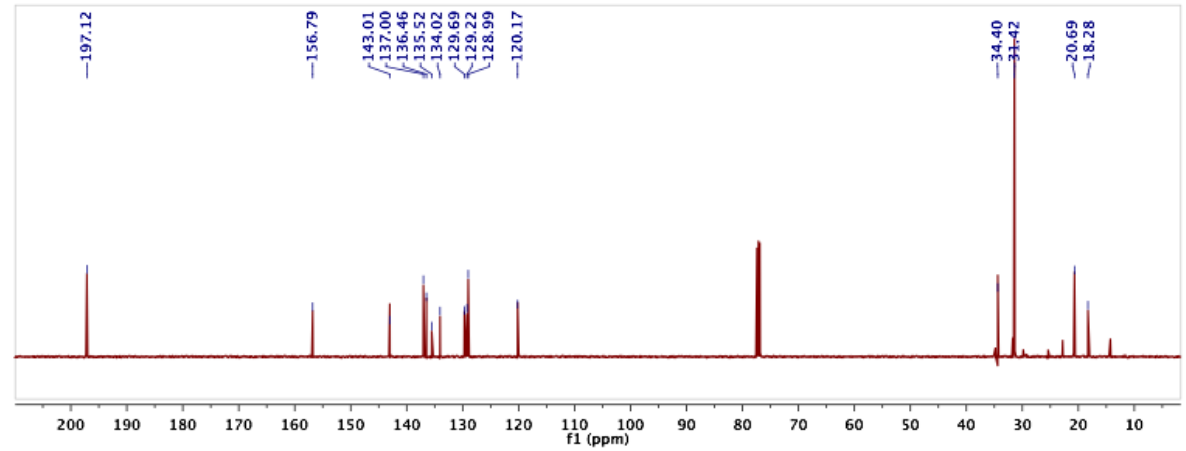


Figure S18. ^{1}H NMR spectrum of **9-s** in $CDCl_3$.

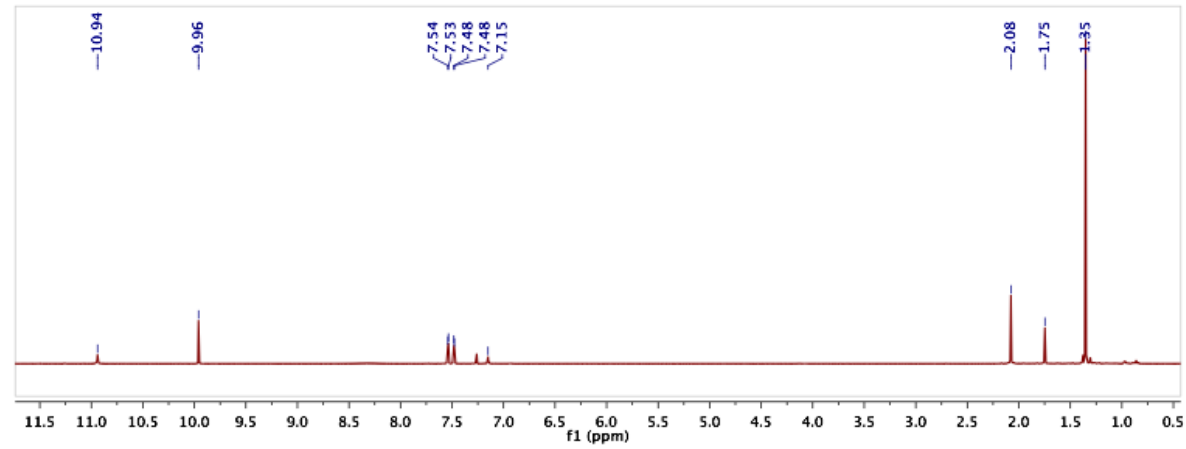


Figure S19. ^{13}C NMR spectrum of **9-s** in $CDCl_3$.

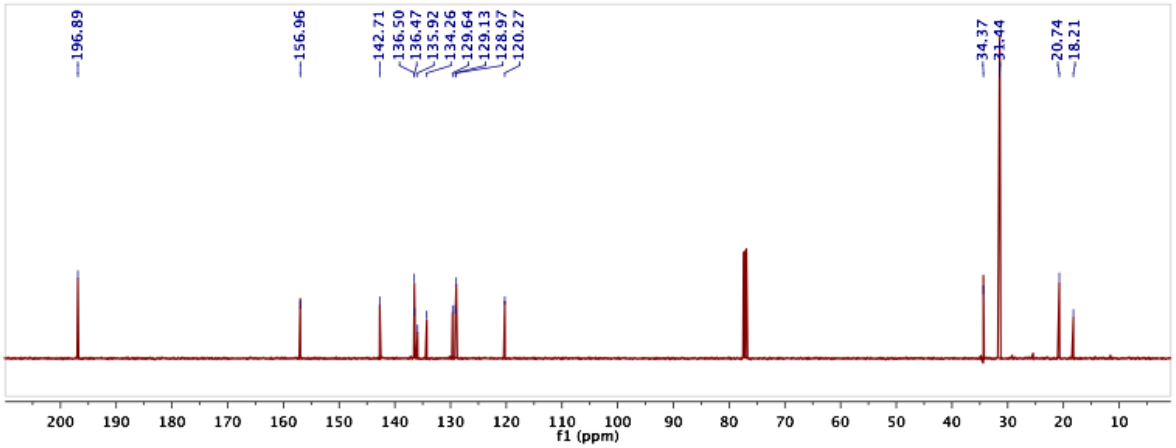


Figure S20. ^{1}H NMR spectrum of **9-s-OMe** in $CDCl_3$.

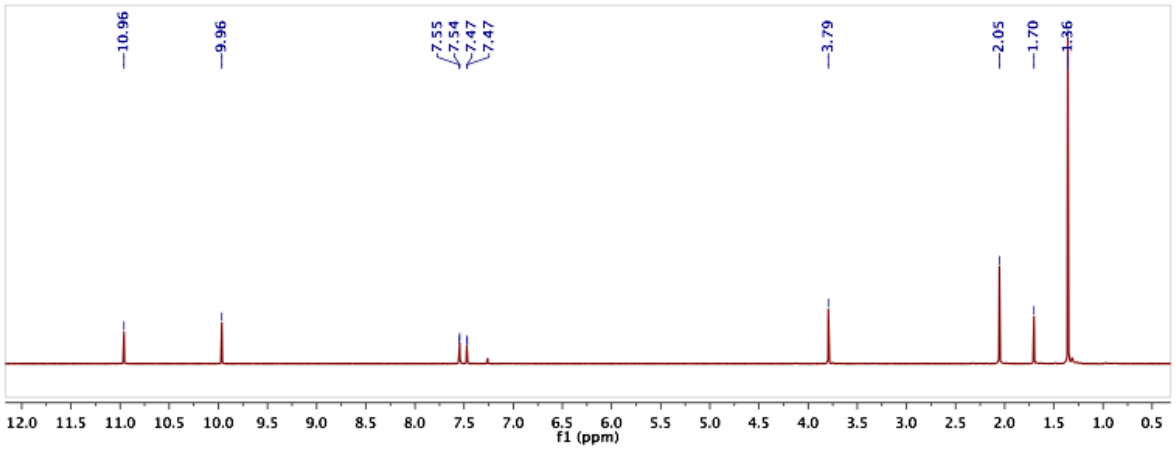


Figure S21. ^{13}C NMR spectrum of **9-s-OMe** in $CDCl_3$.

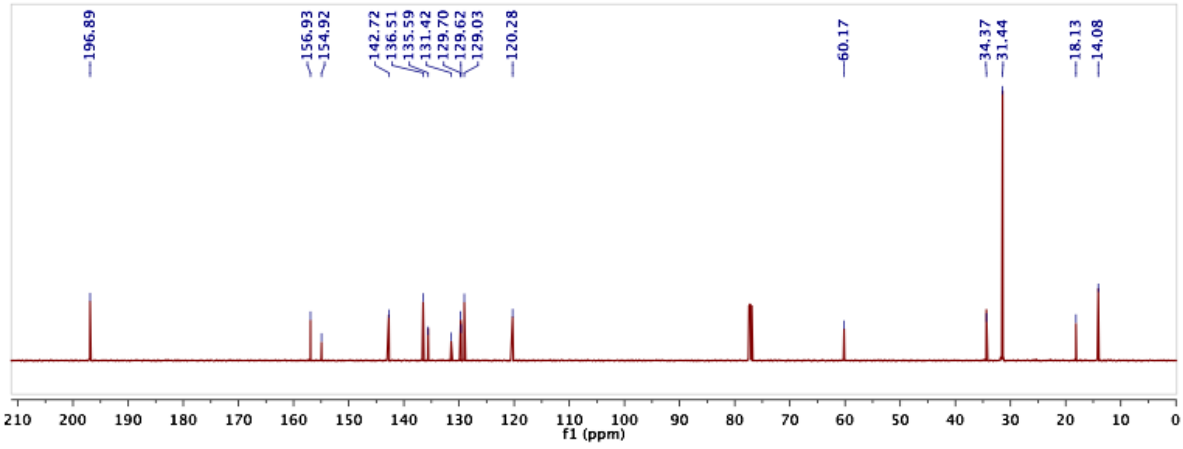


Figure S22. ^{1}H NMR spectrum of **10-a** in $CDCl_3$.

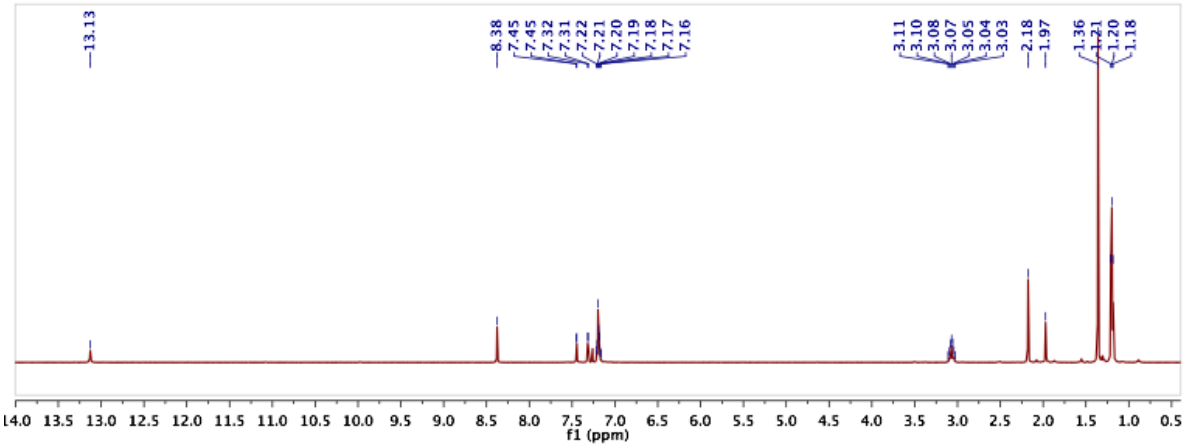


Figure S23. ^{13}C NMR spectrum of **10-a** in $CDCl_3$.

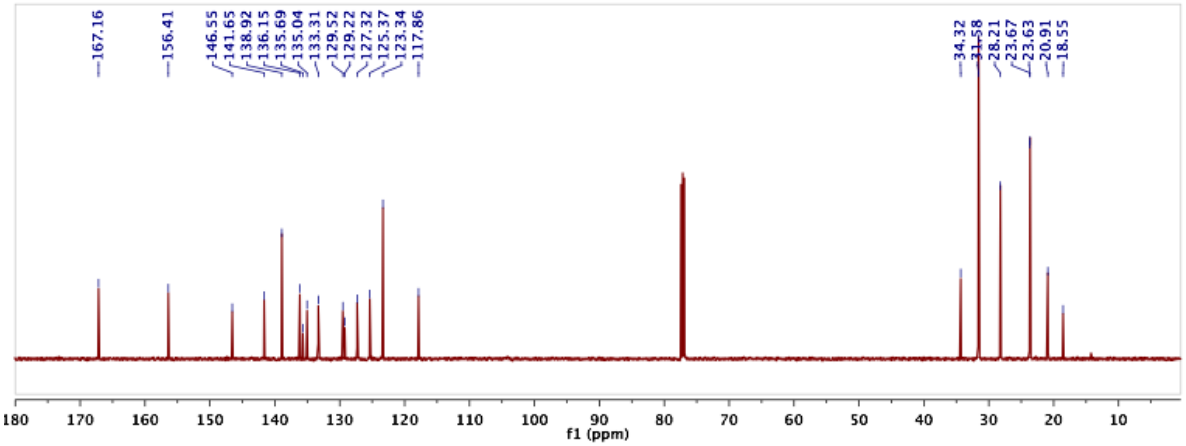


Figure S24. ^{1}H NMR spectrum of **10-s** in $CDCl_3$.

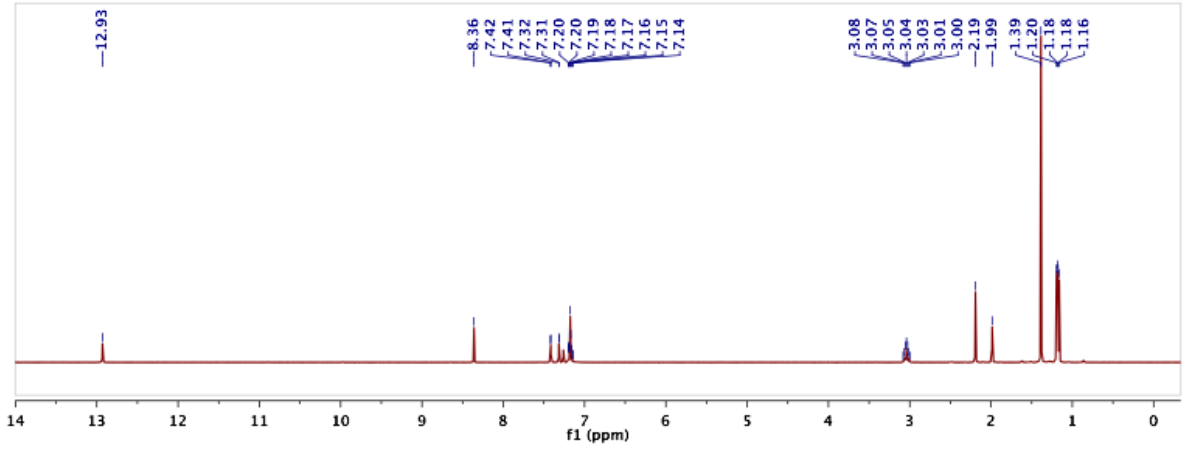


Figure S25. ^{13}C NMR spectrum of **10-s** in $CDCl_3$.

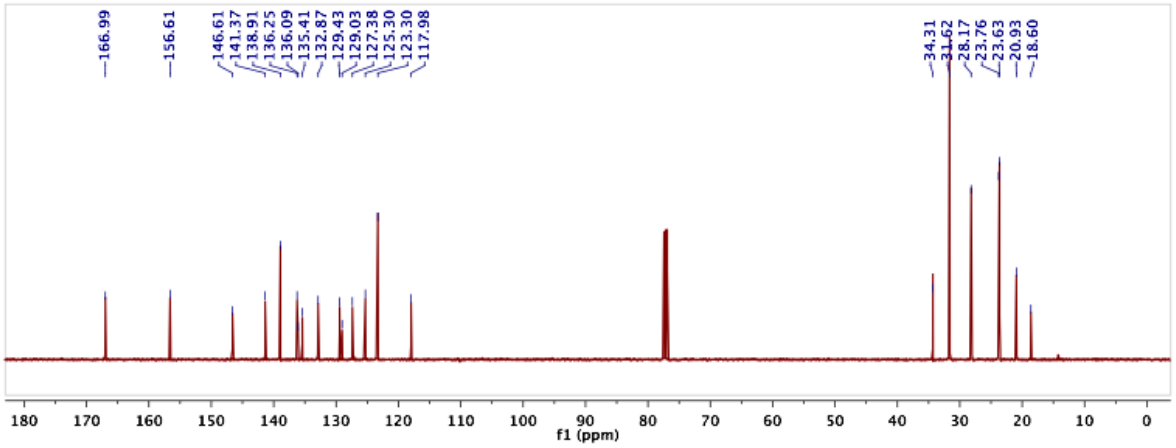


Figure S26. ^{1}H NMR spectrum of **10-s-OMe** in C_6D_6.

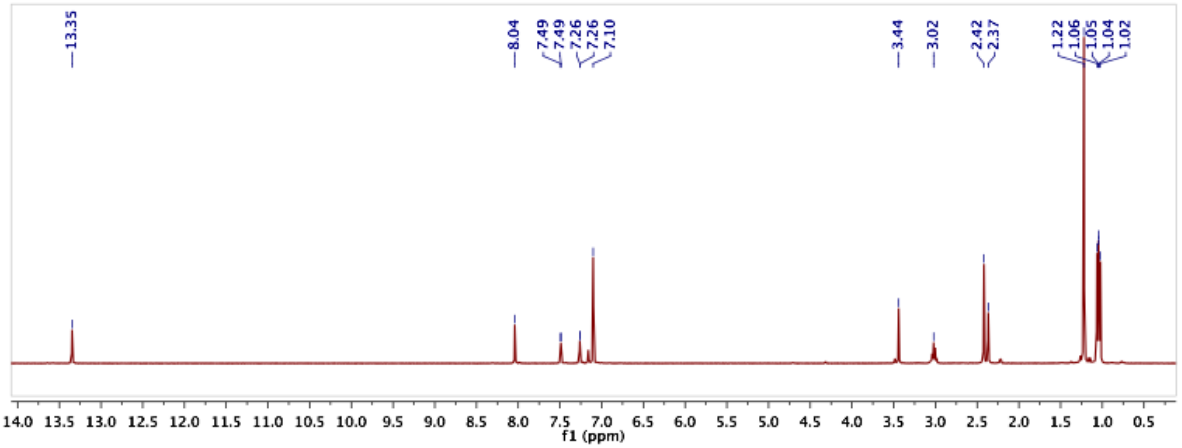


Figure S27. ^{13}C NMR spectrum of **10-s-OMe** in C_6D_6.

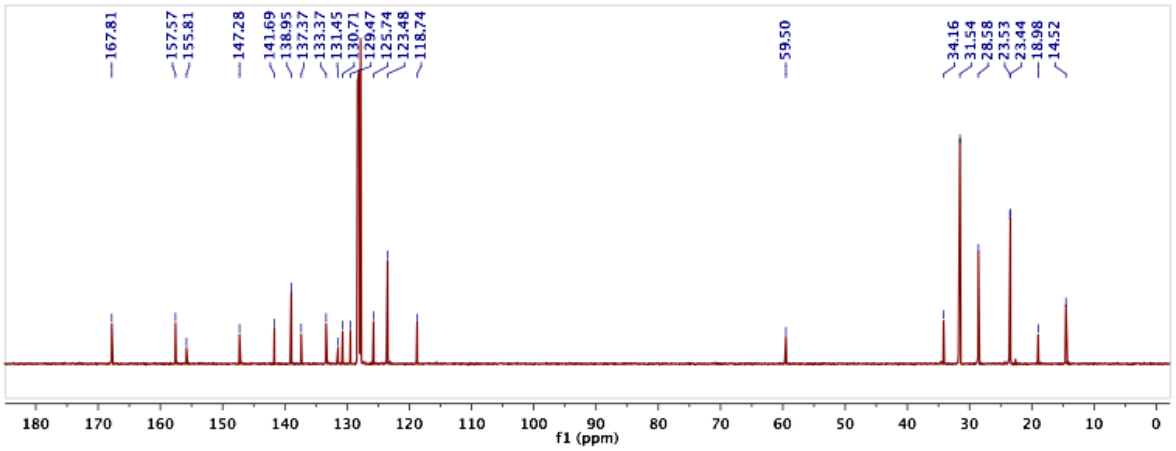


Figure S28. ^{1}H NMR spectrum of the deprotection of **6-a** in $CDCl_3$.

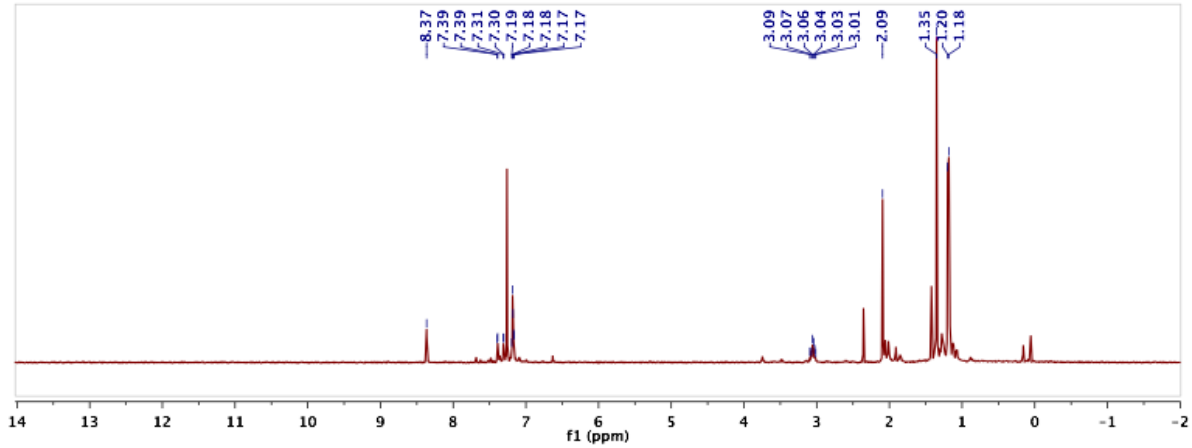


Figure S29. ^{1}H NMR spectrum of the deprotection of **6-s** in $CDCl_3$.

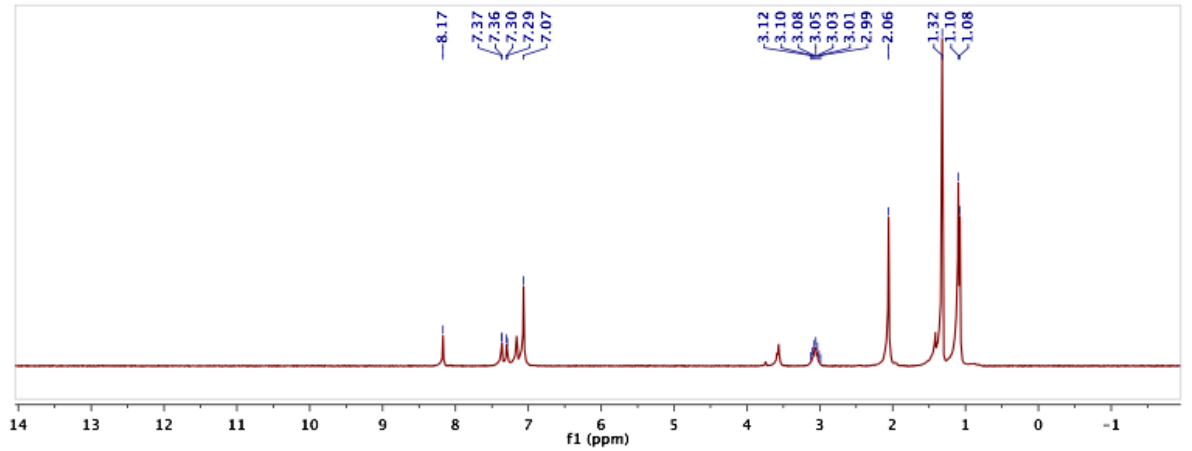


Figure S30. ^{1}H NMR spectrum of **1-a** in C_6D_6.

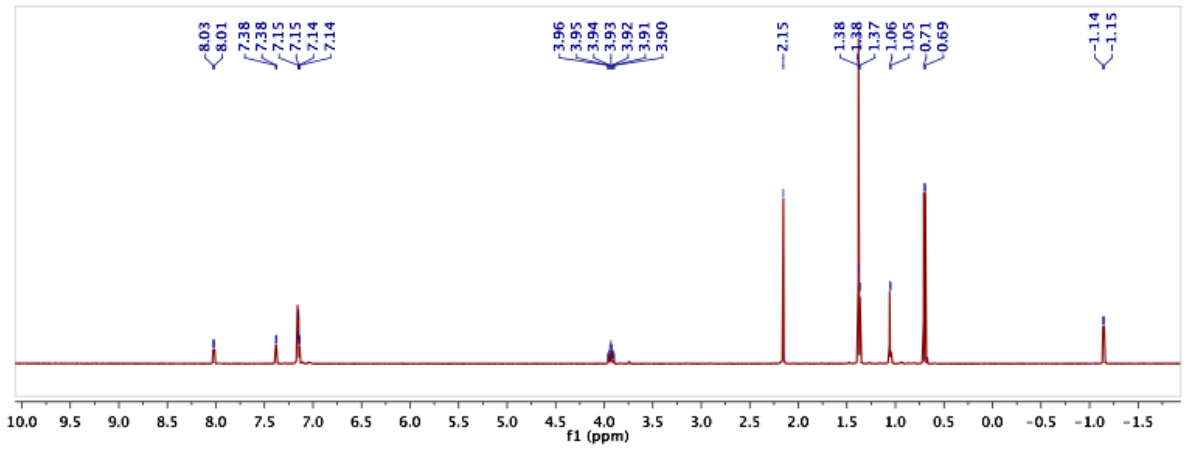


Figure S31. ^{31}P NMR spectrum of **1-a** in C_6D_6.

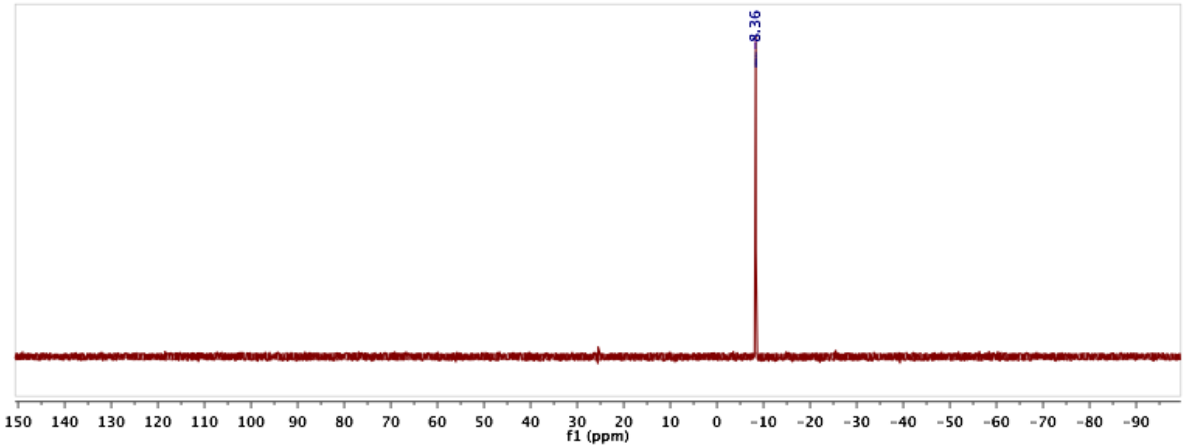


Figure S32. ^{13}C NMR spectrum of **1-a** in C_6D_6.

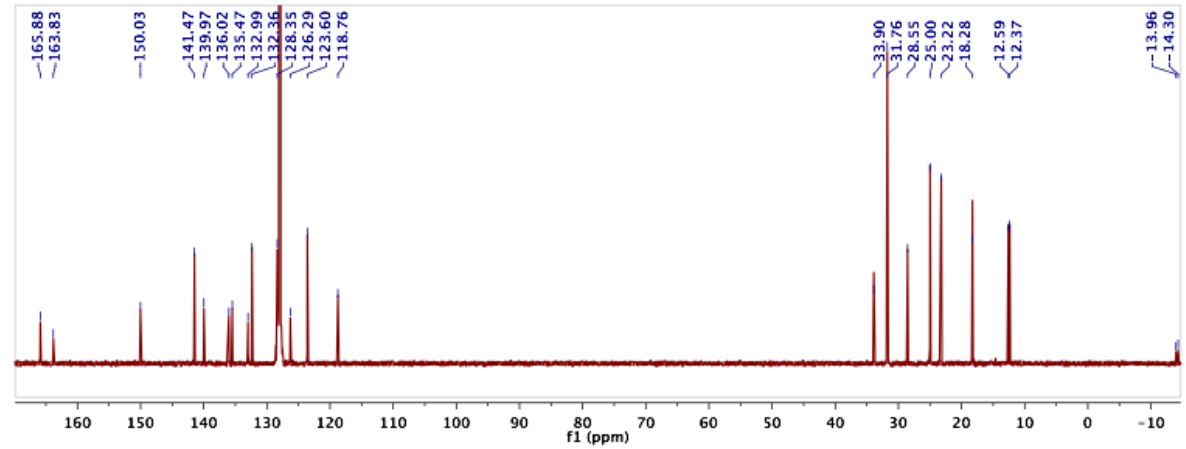


Figure S33. ^{1}H NMR spectrum of **1-s** in C_6D_6.

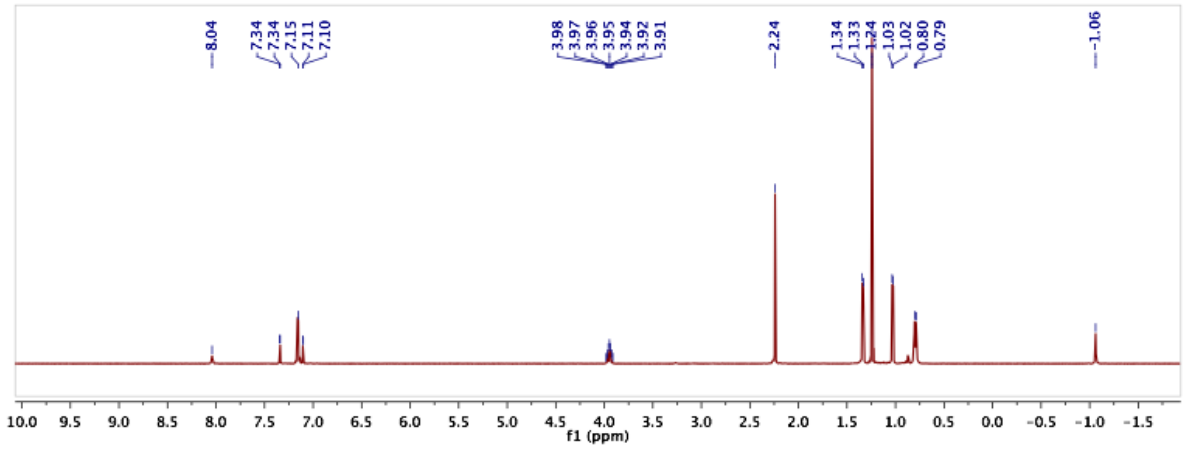


Figure S34. ^{31}P NMR spectrum of **1-s** in C_6D_6.

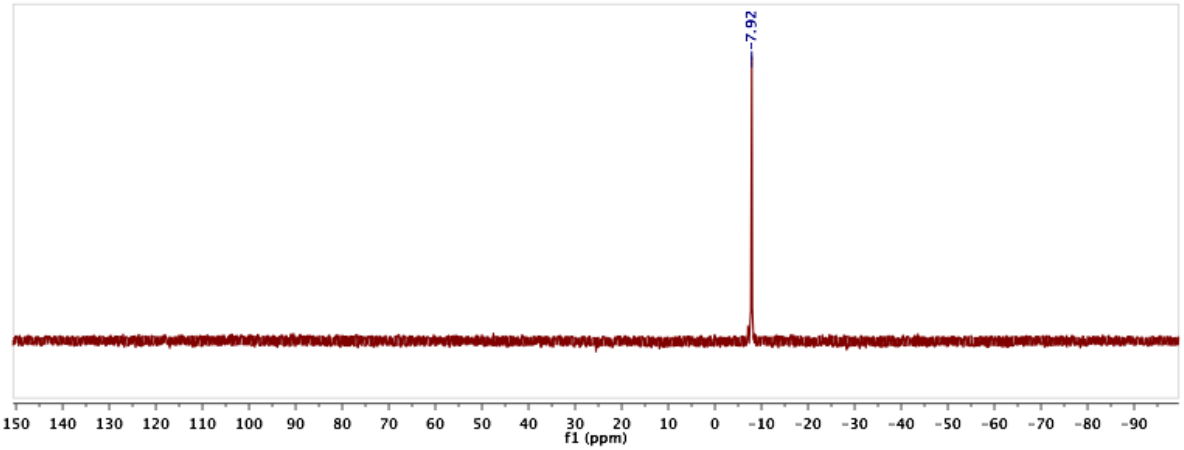


Figure S35. ^{13}C NMR spectrum of **1-s** in C_6D_6.

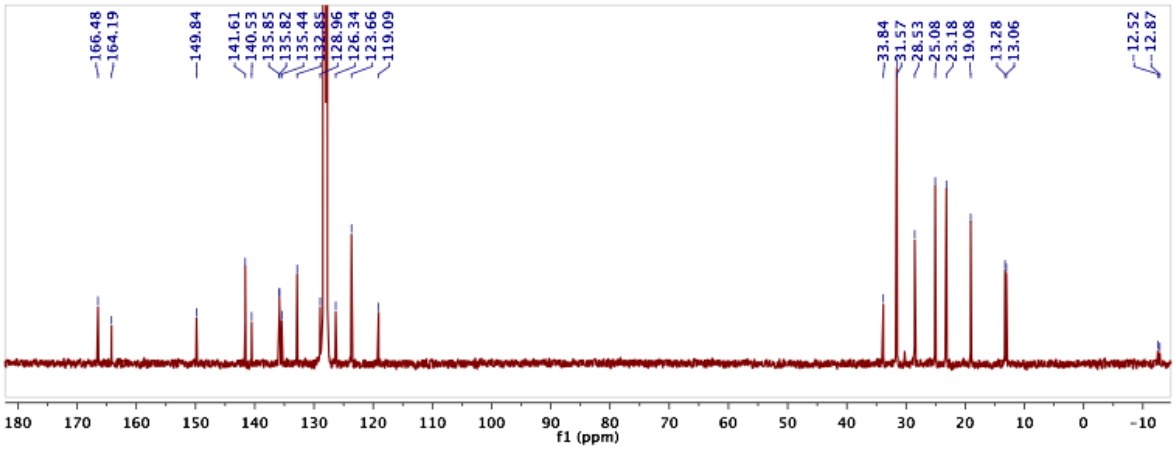


Figure S36. ^{1}H NMR spectrum of **2-a** in C_6D_6.

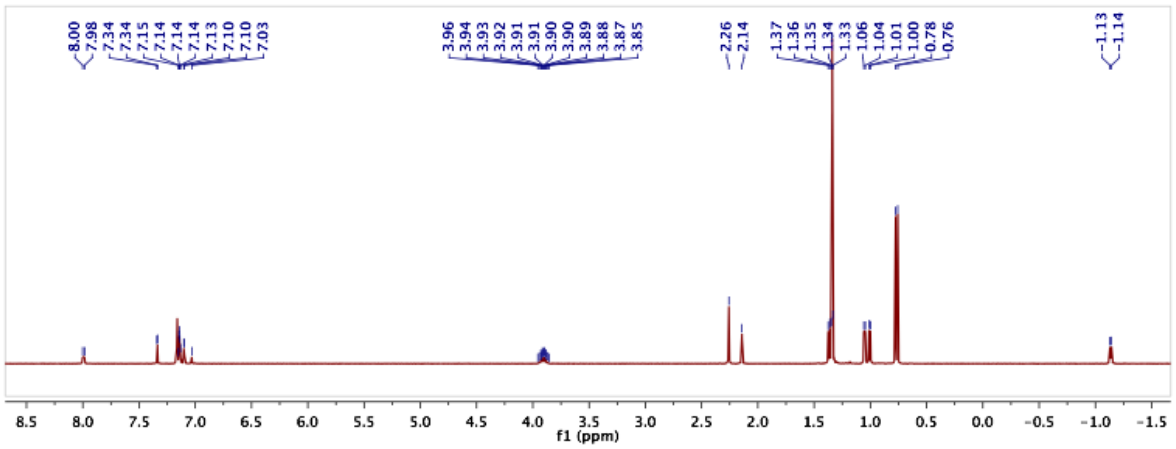


Figure S37. ^{31}P NMR spectrum of **2-a** in C_6D_6.

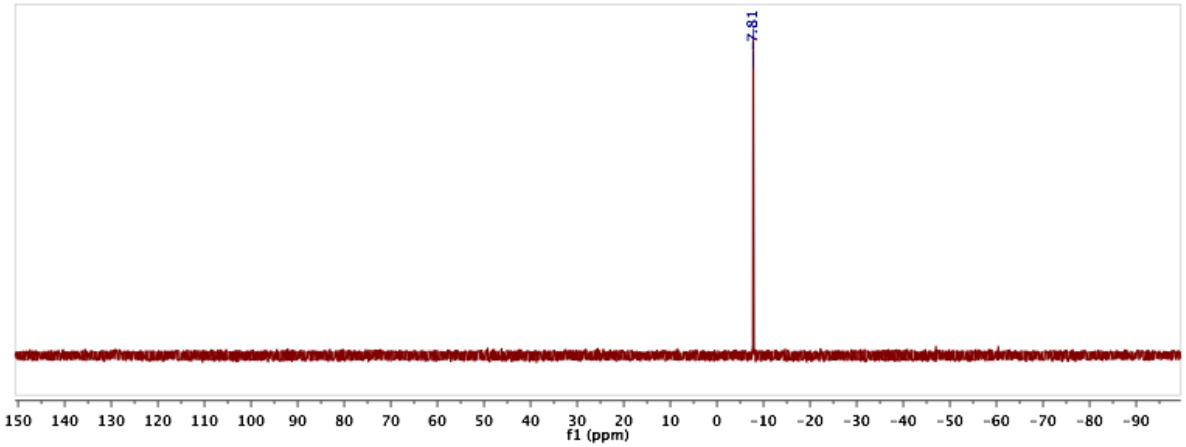


Figure S38. ^{13}C NMR spectrum of **2-a** in C_6D_6.

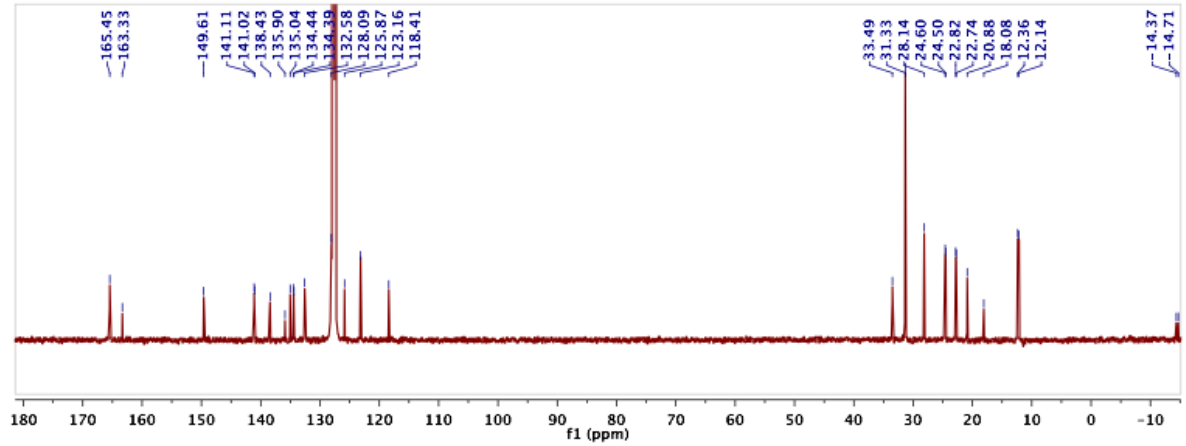


Figure S39. ^{1}H NMR spectrum of **2-s** in C_6D_6.

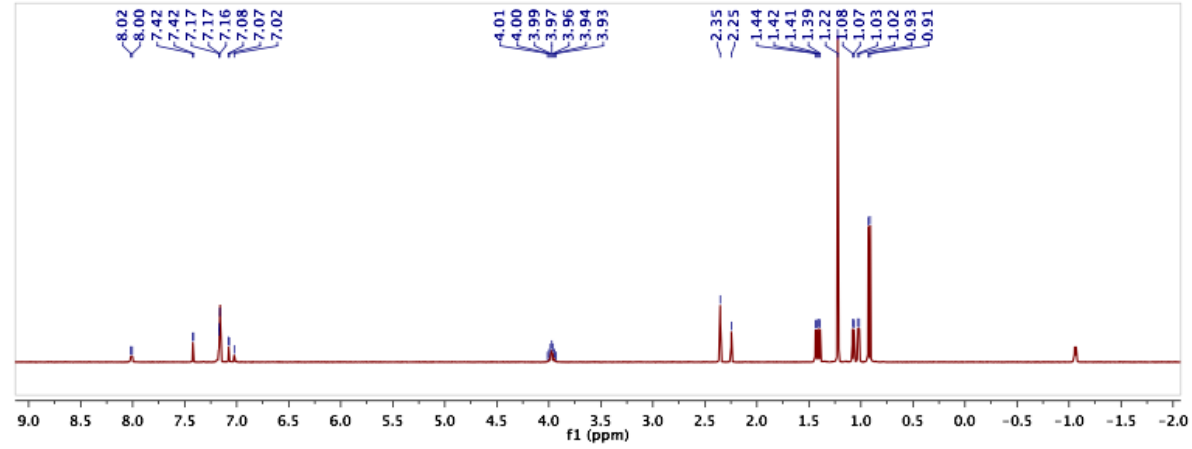


Figure S40. ^{31}P NMR spectrum of **2-s** in C_6D_6.

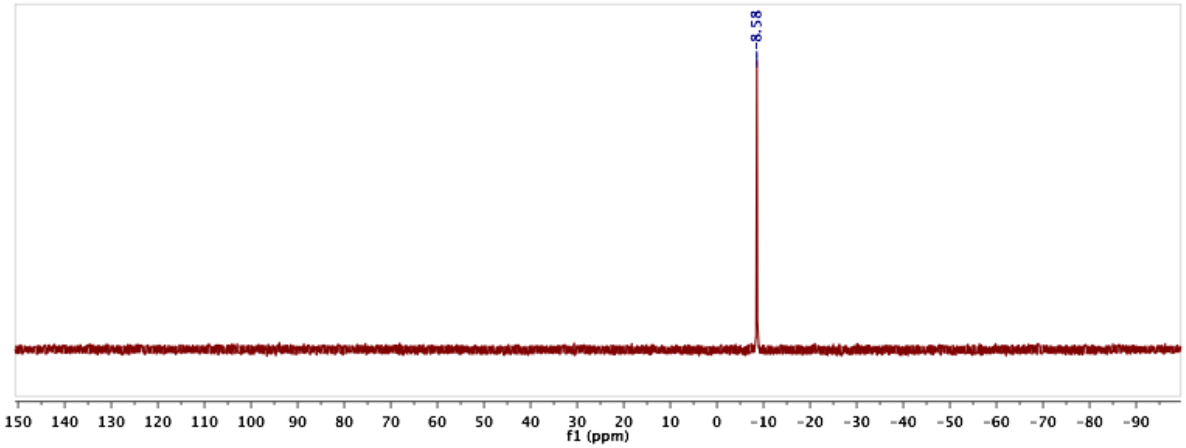


Figure S41. ^{13}C NMR spectrum of **2-s** in C_6D_6.

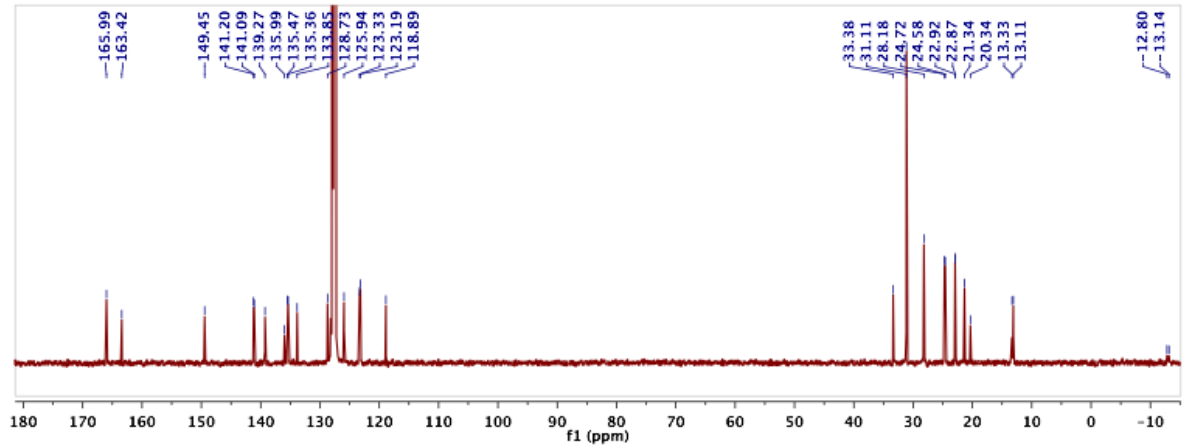


Figure S42. ^{1}H NMR spectrum of **2-s-OMe** in C_6D_6.

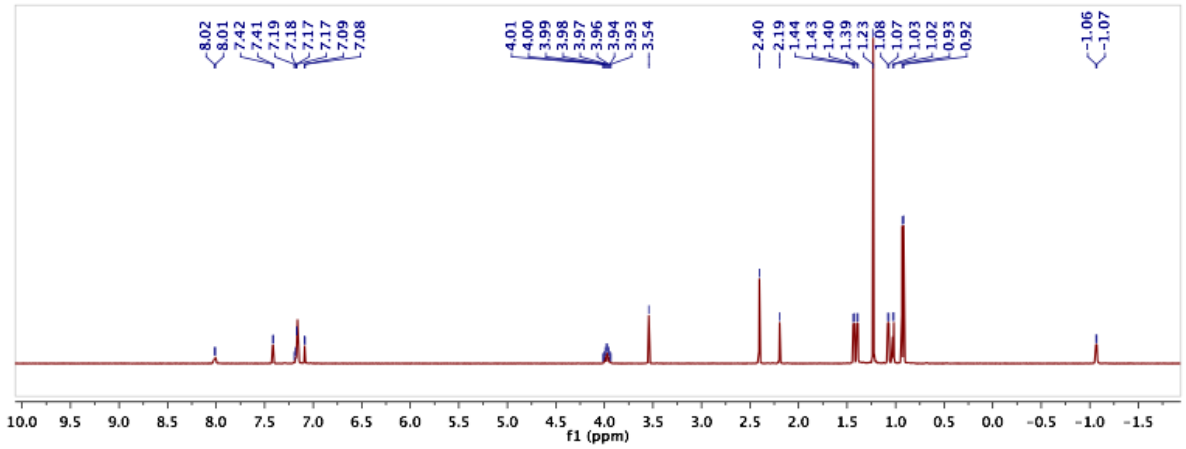


Figure S43. ^{31}P NMR spectrum of **2-s-OMe** in C_6D_6.

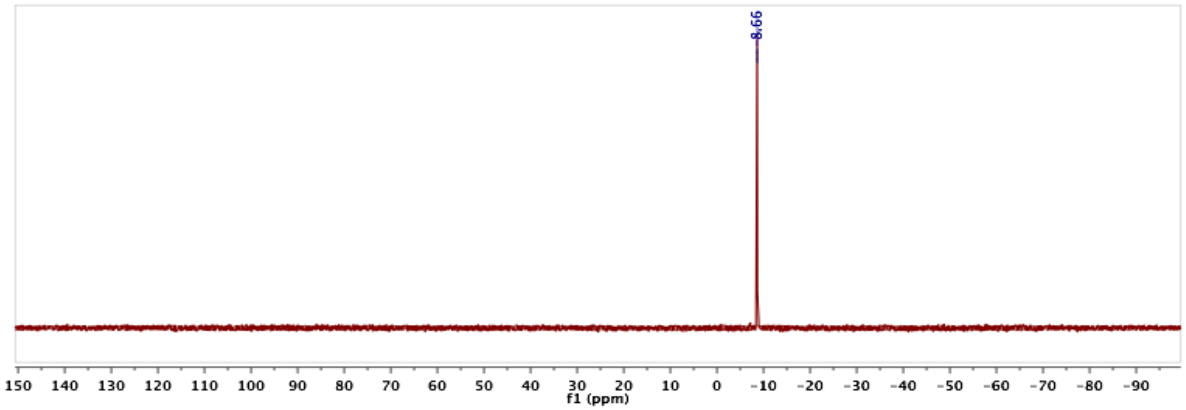


Figure S44. ^{13}C NMR spectrum of **2-s-OMe** in C_6D_6.

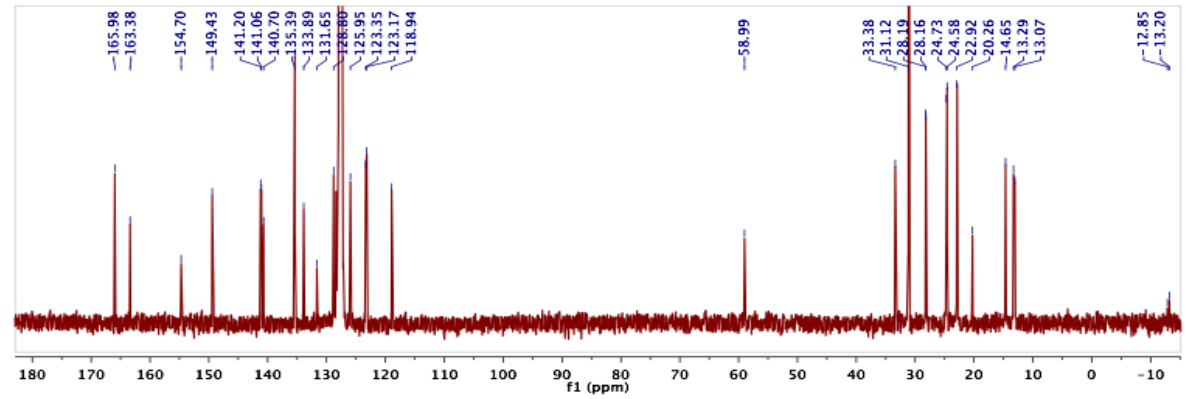


Figure S45. ^{1}H-^{1}H NOESY spectrum of **2-s-OMe** in C_6D_6 (no cross peaks between the PMe_3 and any aryl peaks, confirming syn atropisomer).

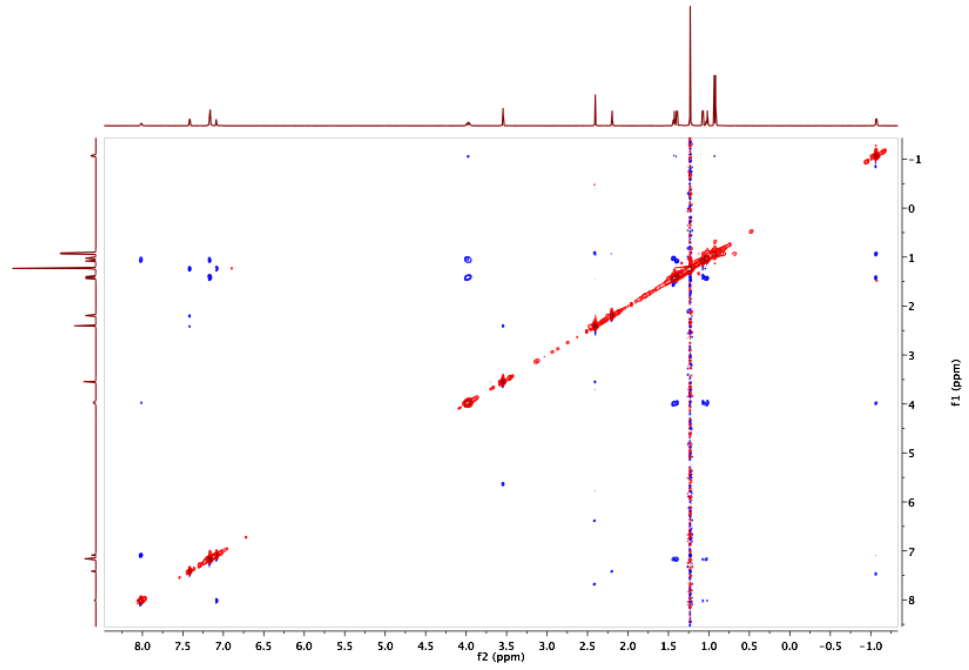


Figure S46. ^{1}H NMR spectrum of **N(allyl)(^{n}Pr)$_2$** in $CDCl_3$.

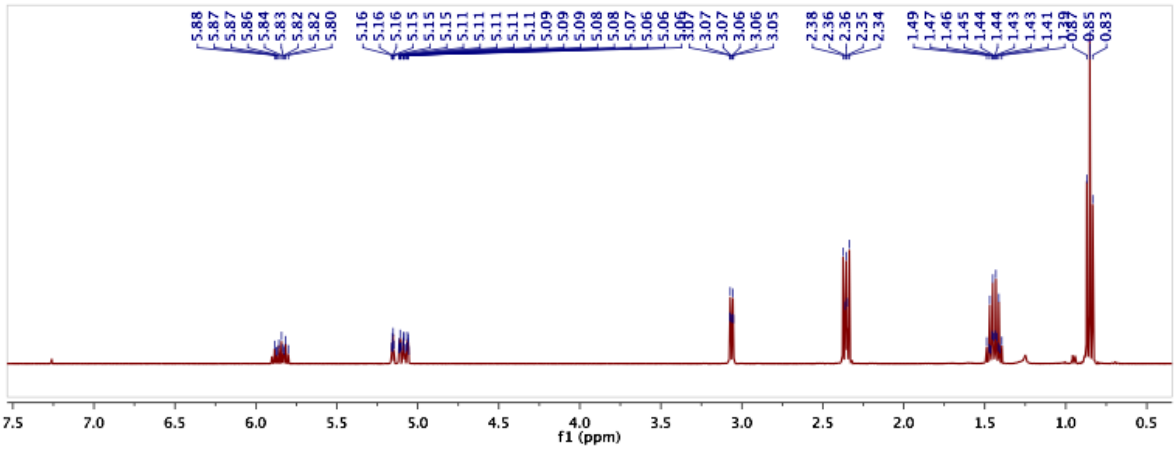


Figure S47. ^{13}C NMR spectrum of **N(allyl)(^{n}Pr)$_2$** in $CDCl_3$.

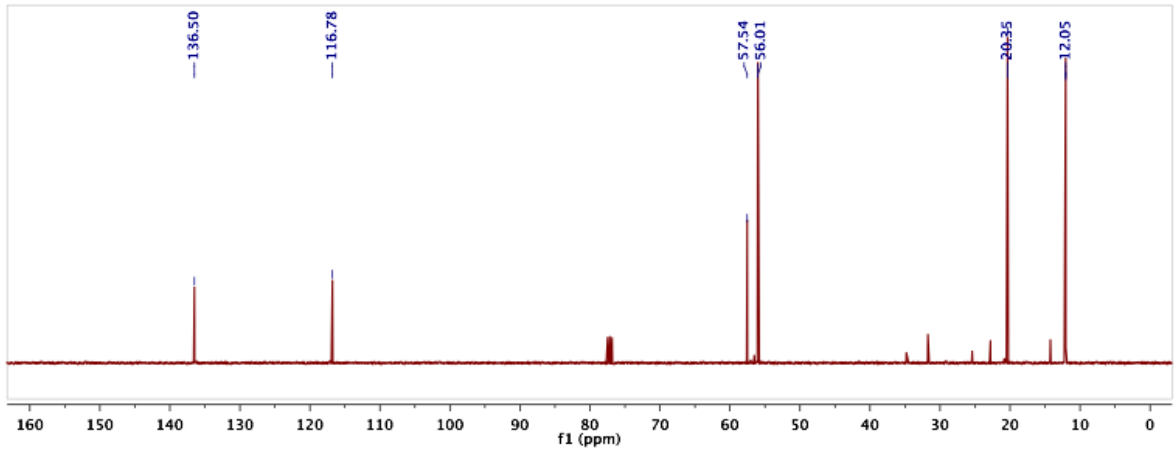


Figure S48. ^{1}H NMR spectrum of **N(butenyl)(^{n}Pr)$_2$** in $CDCl_3$.

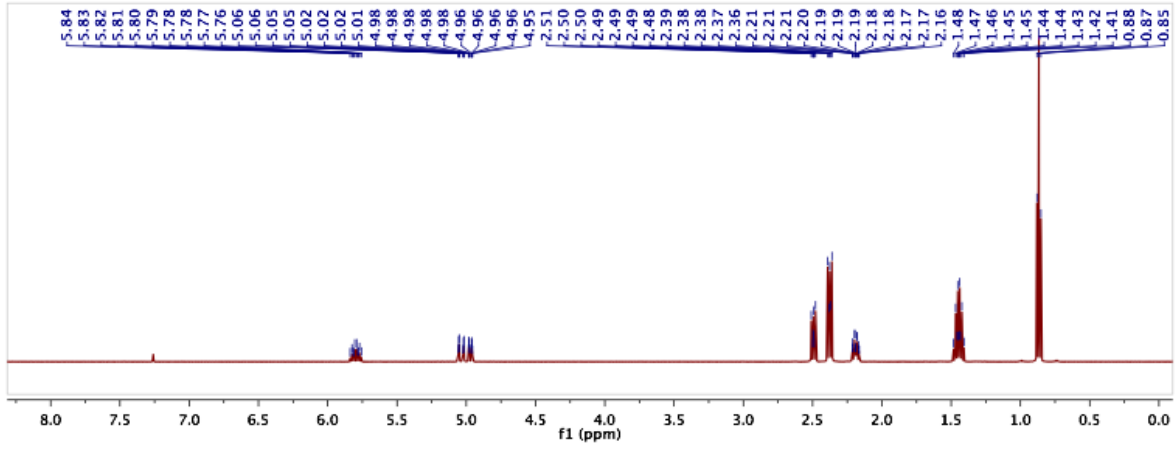


Figure S49. ^{13}C NMR spectrum of **N(butenyl)(″Pr)$_2$** in $CDCl_3$.

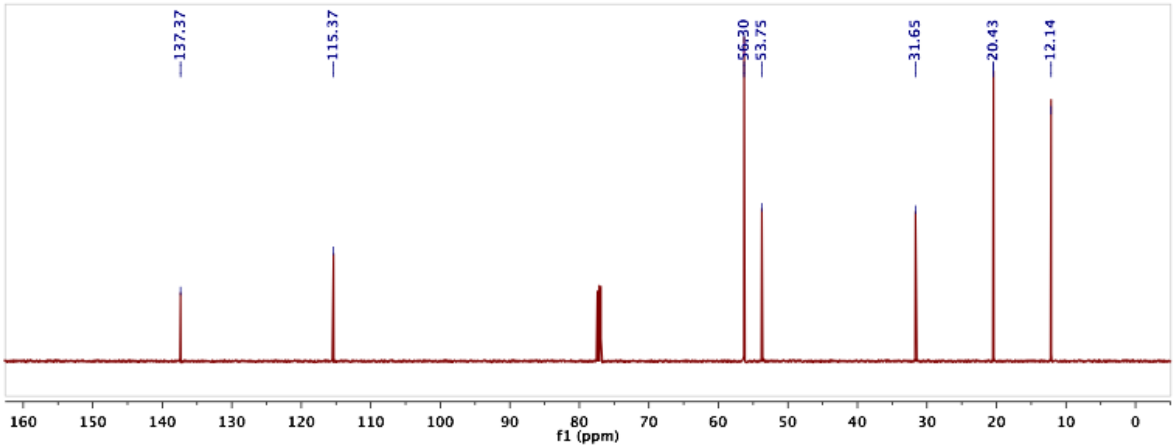


Figure S50. ^{1}H NMR spectrum of **N(pentenyl)(″Pr)$_2$** in $CDCl_3$.

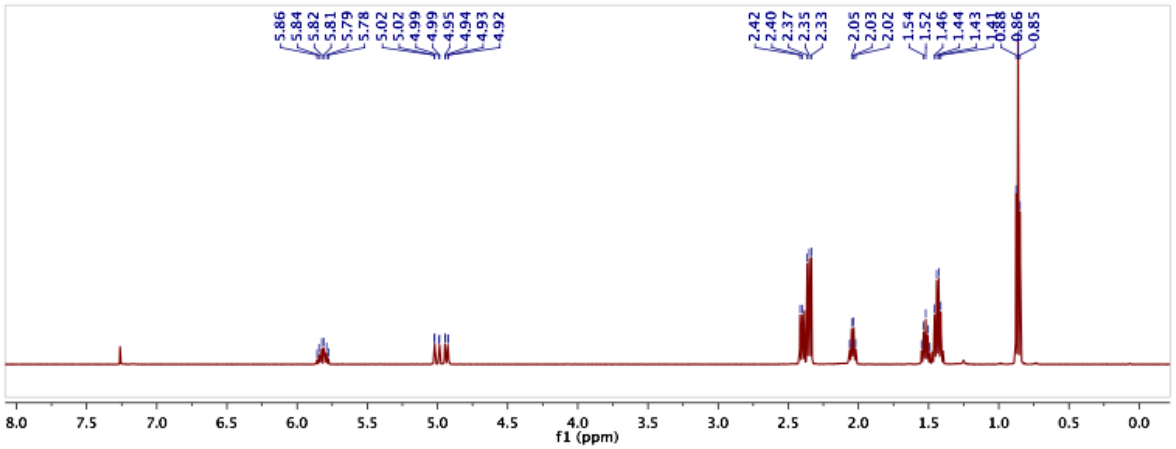


Figure S51. ^{13}C NMR spectrum of **N(pentenyl)(″Pr)$_2$** in $CDCl_3$.

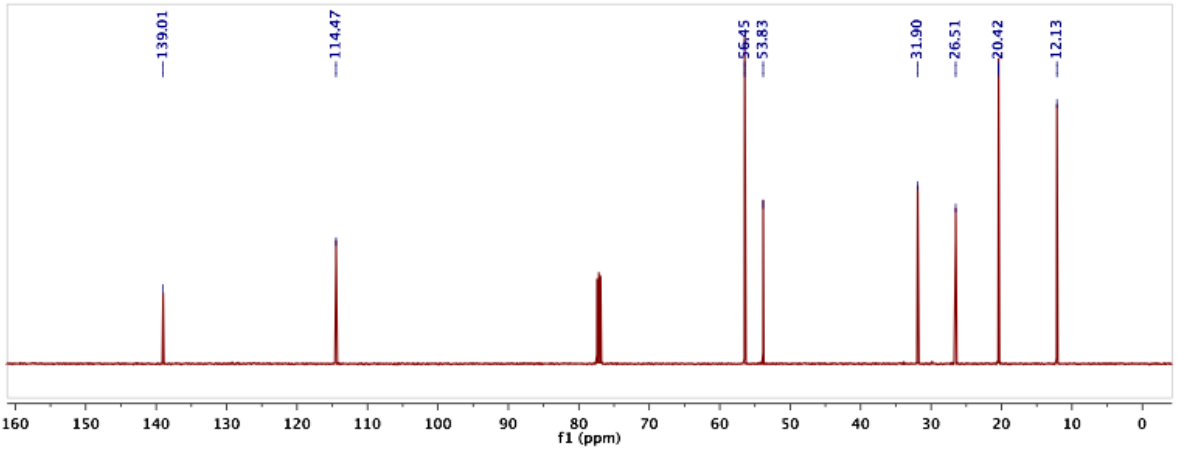


Figure S52. ^{1}H NMR spectrum of **N(hexenyl)(^{n}Pr)$_2$** in $CDCl_3$.

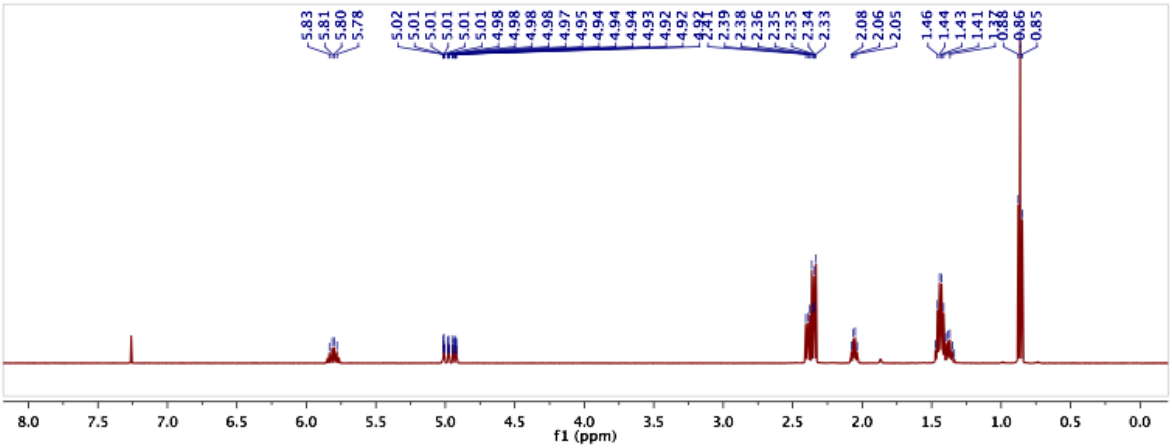


Figure S53. ^{13}C NMR spectrum of **N(hexenyl)(^{n}Pr)$_2$** in $CDCl_3$.

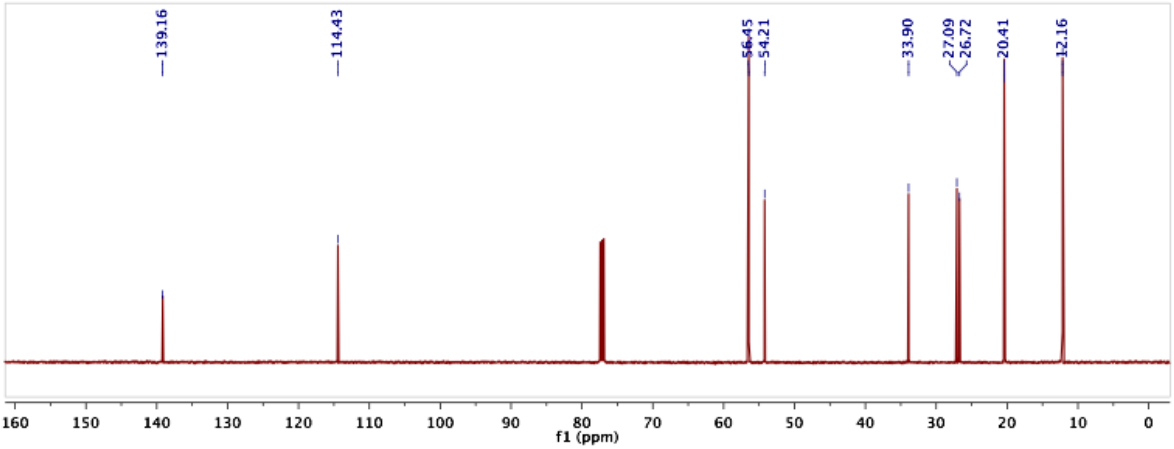


Figure S54. ^{1}H NMR spectrum of **N(heptenyl)(^{n}Pr)$_2$** in $CDCl_3$.

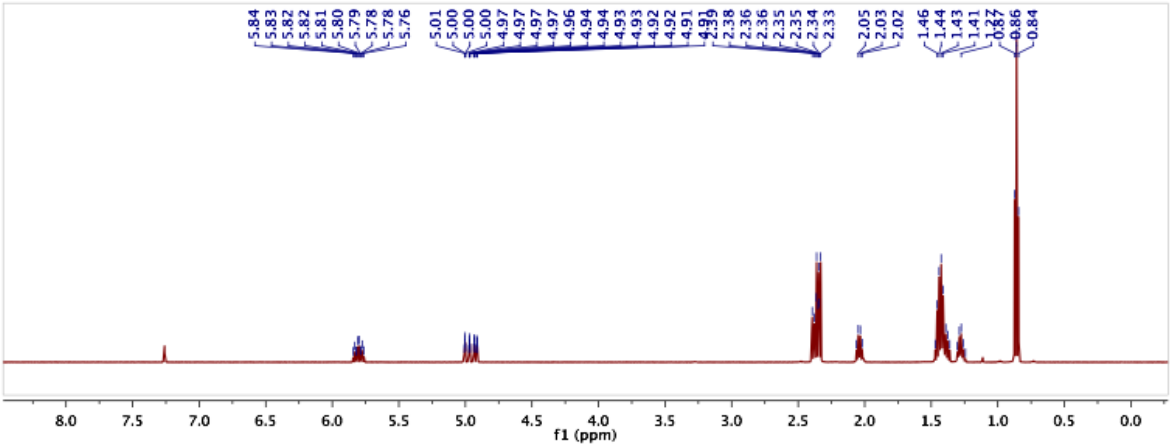


Figure S55. ^{13}C NMR spectrum of **N(heptenyl)(″Pr)$_2$** in $CDCl_3$.

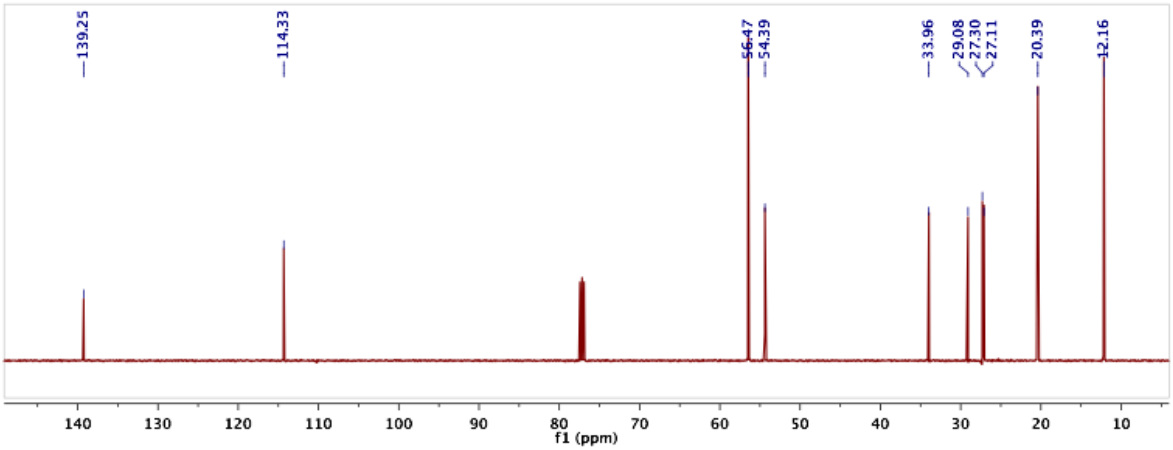


Figure S56. ^{1}H NMR spectrum of **N(octenyl)(″Pr)$_2$** in $CDCl_3$.

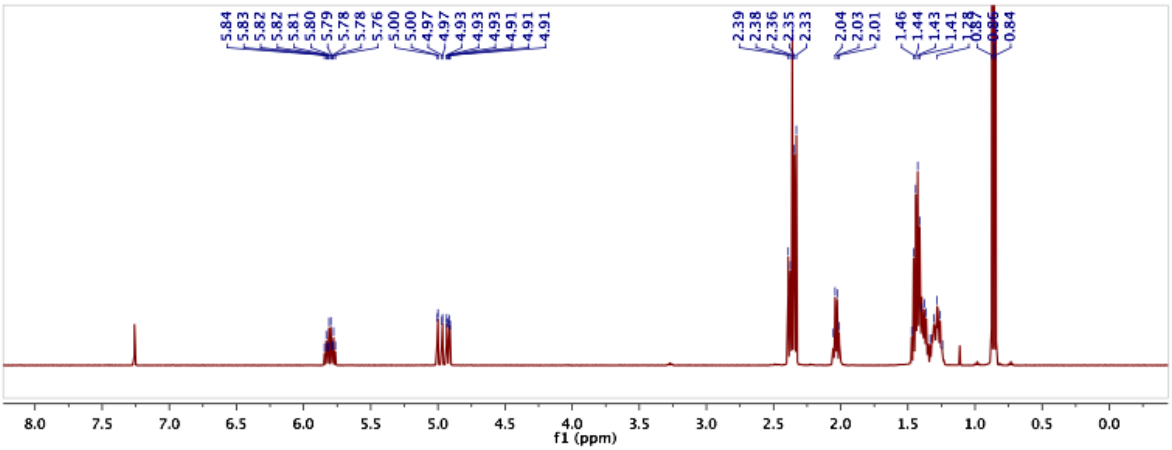


Figure S57. ^{13}C NMR spectrum of **N(octenyl)(″Pr)$_2$** in $CDCl_3$.

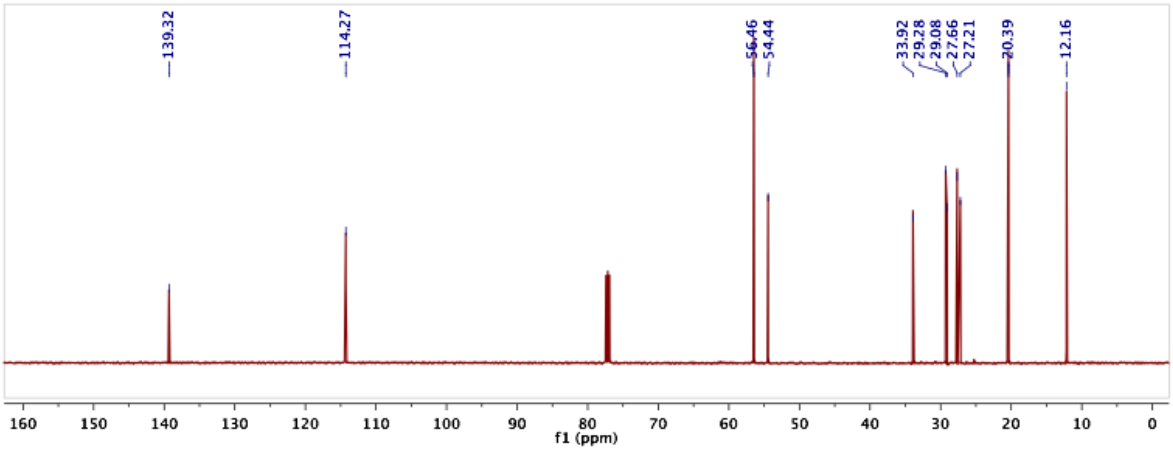


Figure S58. ^{1}H NMR spectrum of **N(pentenyl)(Et)$_2$** in $CDCl_3$.

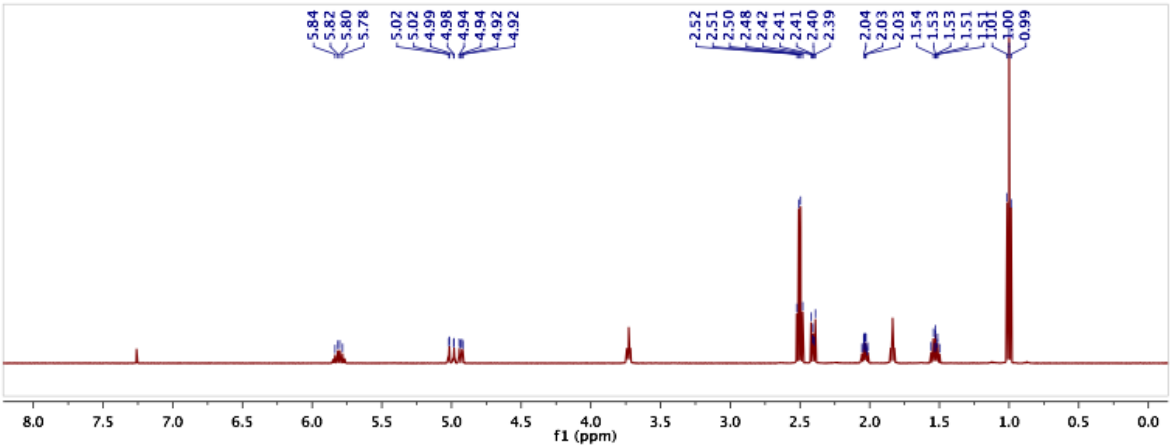
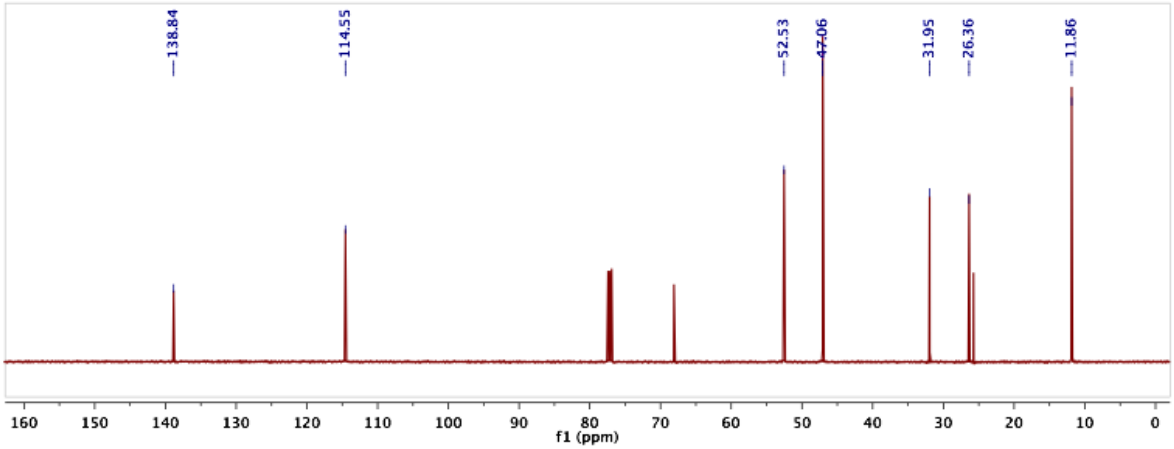


Figure S59. ^{13}C NMR spectrum of **N(pentenyl)(Et)$_2$** in $CDCl_3$.



Representative ^{1}H and ^{13}C NMR spectra of polymers

Figure S60. ^{1}H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **1-s**. The integral of the multiplet at 0.94 ppm was set to 3 for the determination of the extent of branching in the polymer chain.

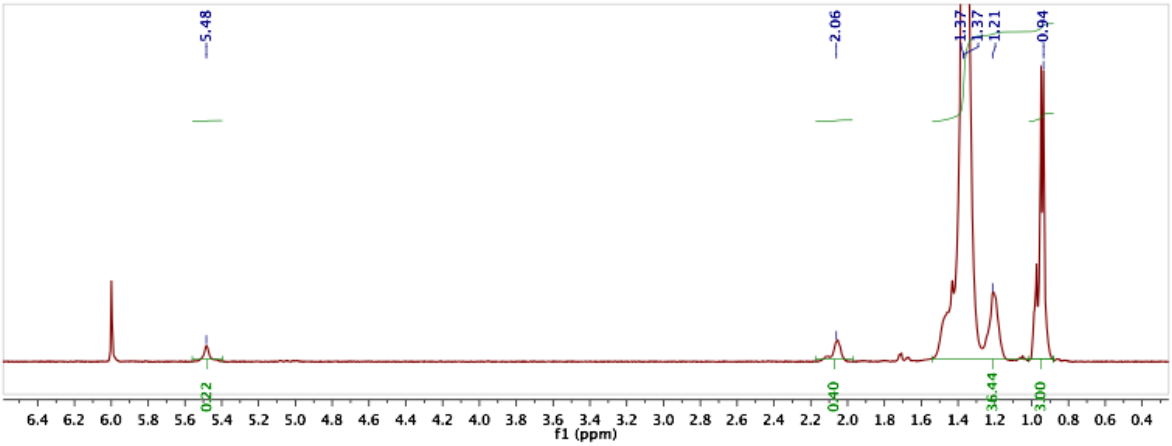


Figure S61. ^{13}C NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **1-s**. The peak at 30 ppm is off scale. The integrated peaks at 37.59 and 38.25 ppm were used to compare the amount of methyl vs butyl branching in the polymer.

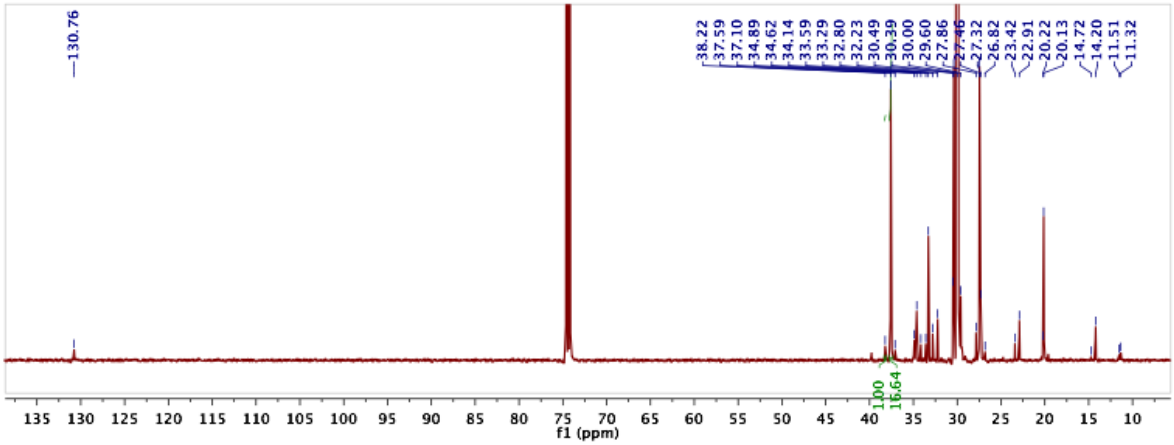


Figure S62. ^{1}H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **2-s**. The integral of the multiplet at 0.93 ppm was set to 3 for the determination of the extent of branching in the polymer chain.

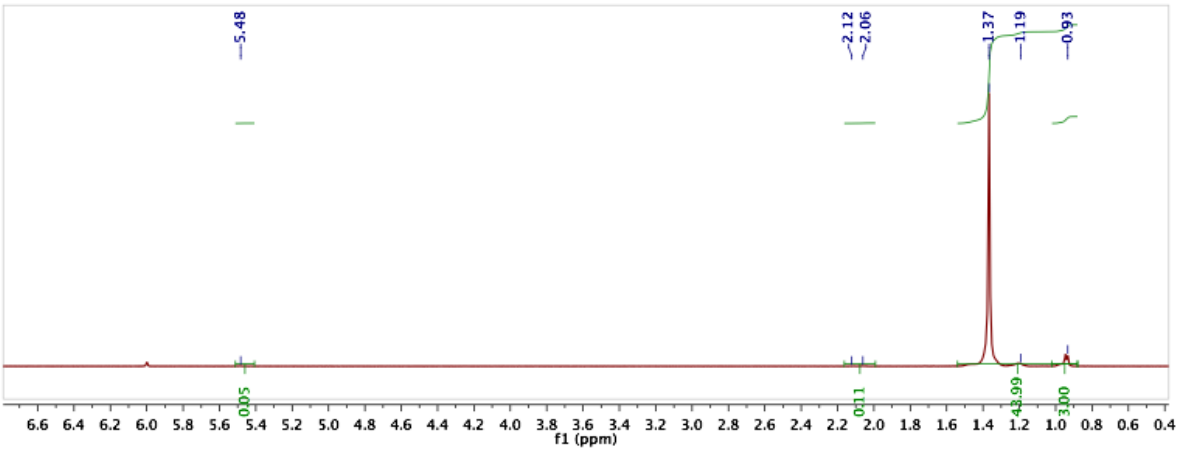


Figure S63. ^{13}C NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **2-s**. The peak at 30 ppm is off scale. The integrated peaks at 37.59 and 38.25 ppm were used to compare the amount of methyl vs butyl branching in the polymer.

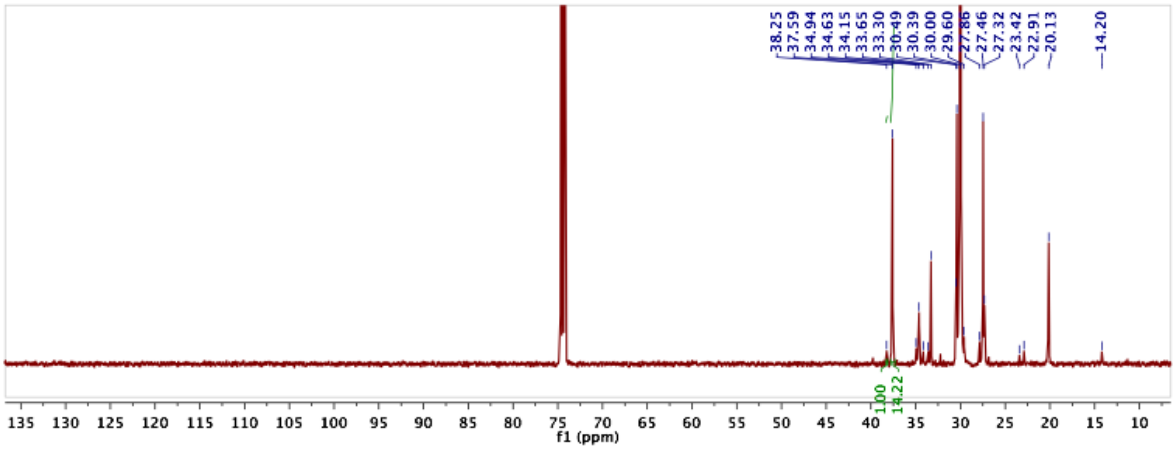


Figure S64. ^{1}H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **1-s** in the presence of NMe(^{n}Pr)$_2$.

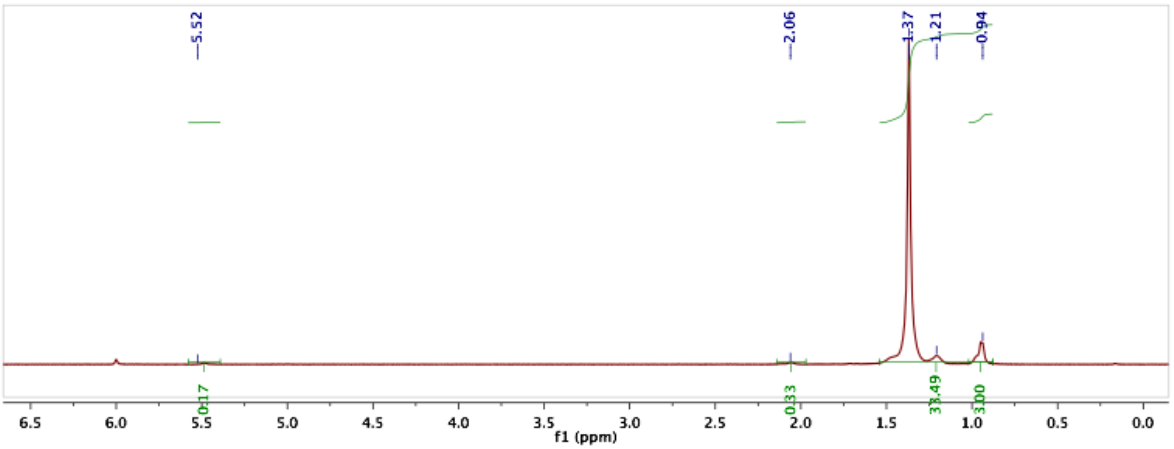


Figure S65. ^{13}C NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **1-s** in the presence of NMe(^{n}Pr)$_2$. The peak at 30 ppm is off scale.

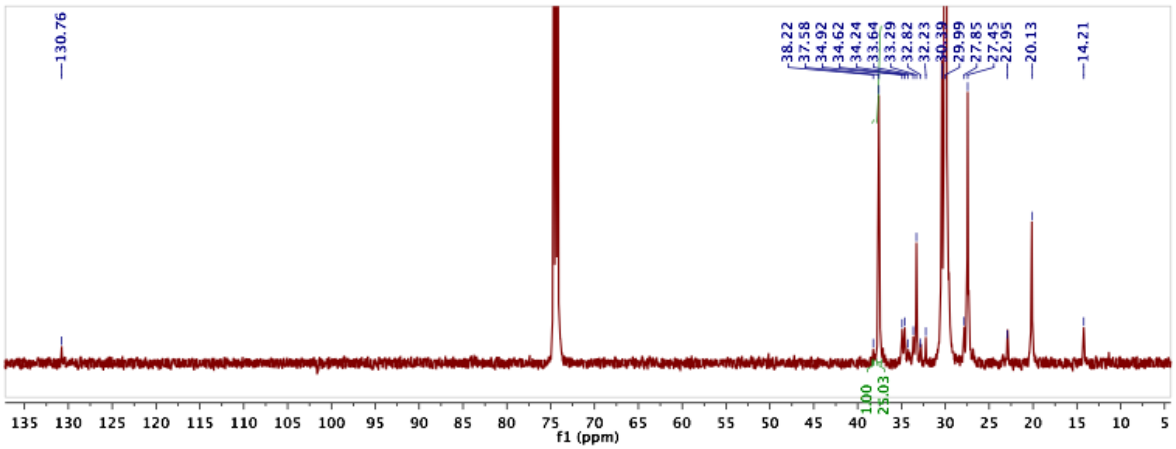


Figure S66. ^{1}H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **2-s** in the presence of NMe(^{n}Pr)$_2$.

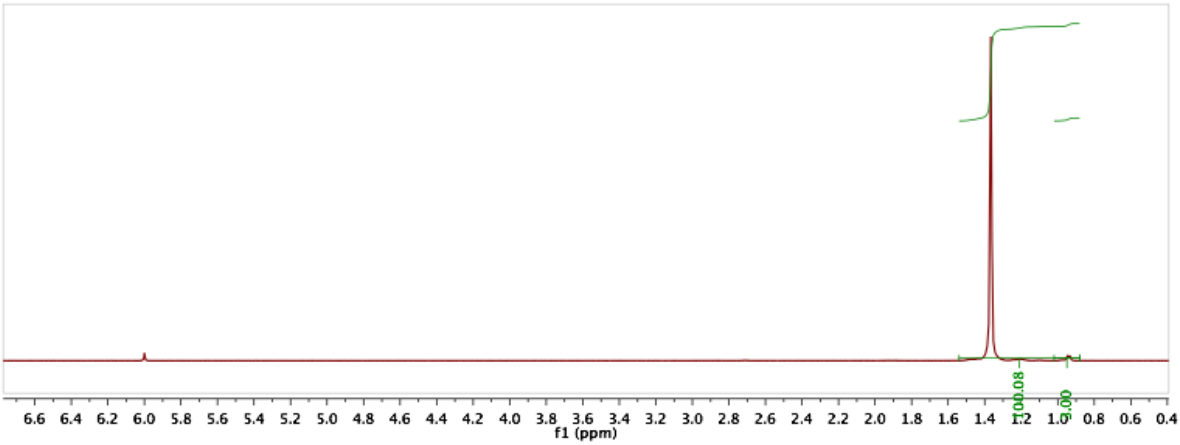


Figure S67. ^{13}C NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **2-s** in the presence of NMe(^{n}Pr)$_2$. The peak at 30 ppm is off scale.

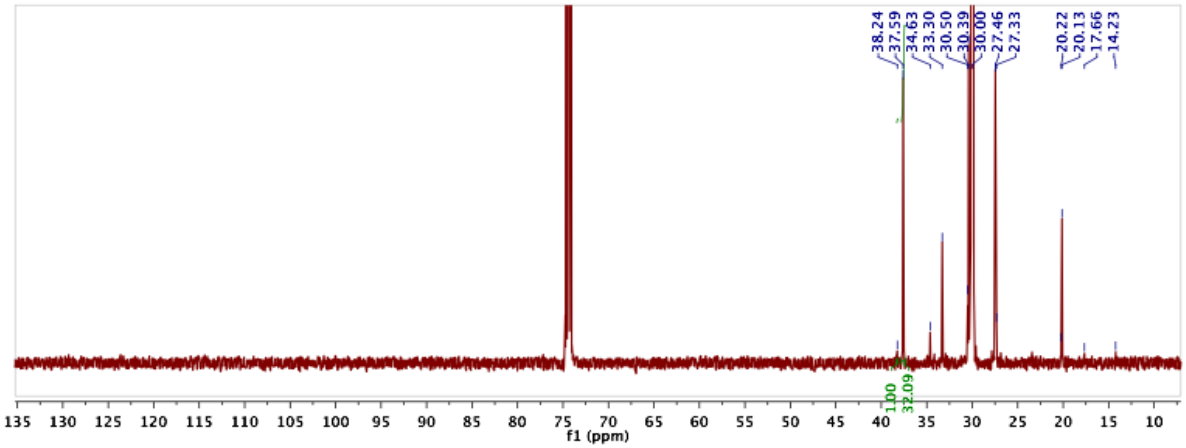


Figure S68. ^1H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **1-s**. The blown up peak (left) at 12.6 ppm is the ammonium proton.

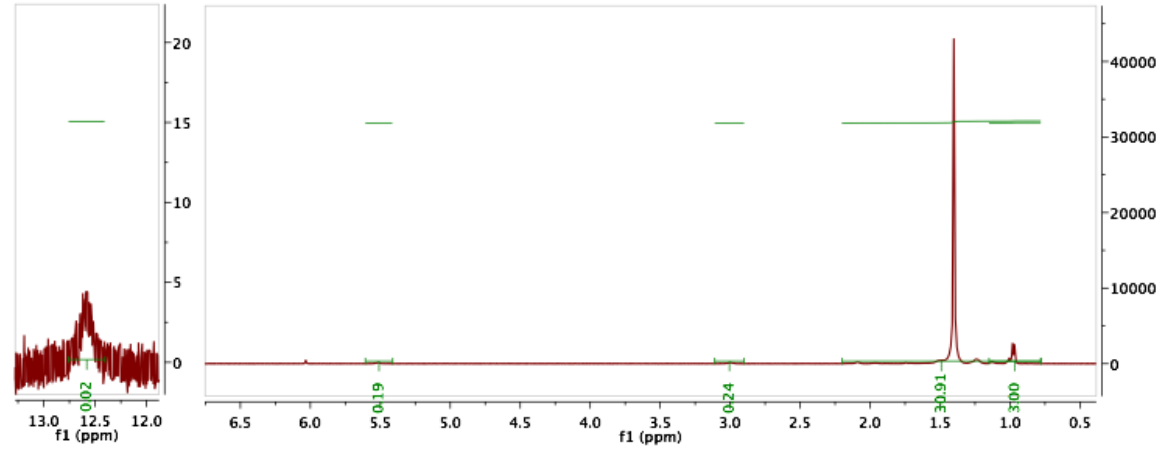


Figure S69. ^{13}C NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **1-s**. The peak at 30 ppm is off scale.

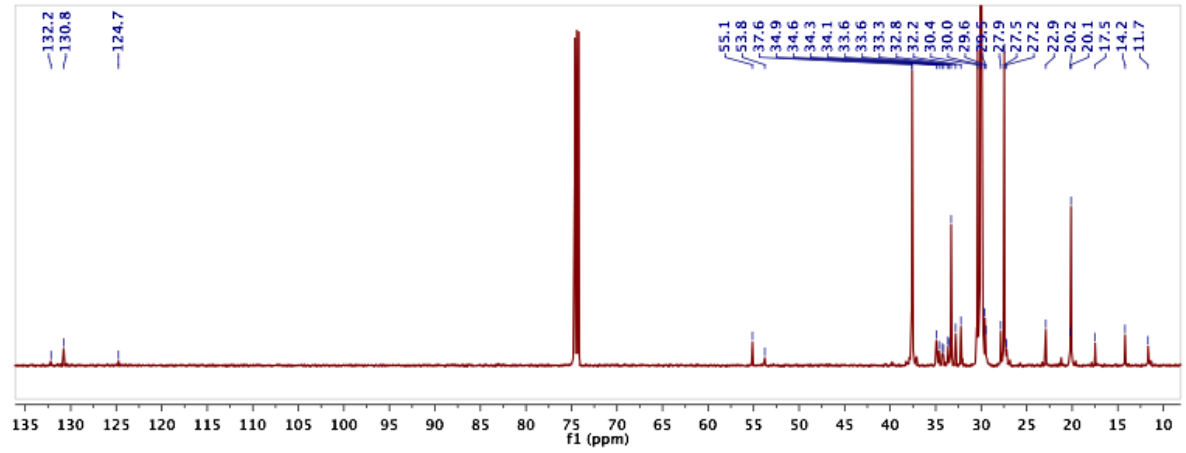


Figure S70. ^{1}H NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **2-s**. The blown up peak (left) at 12.6 ppm is the ammonium proton.

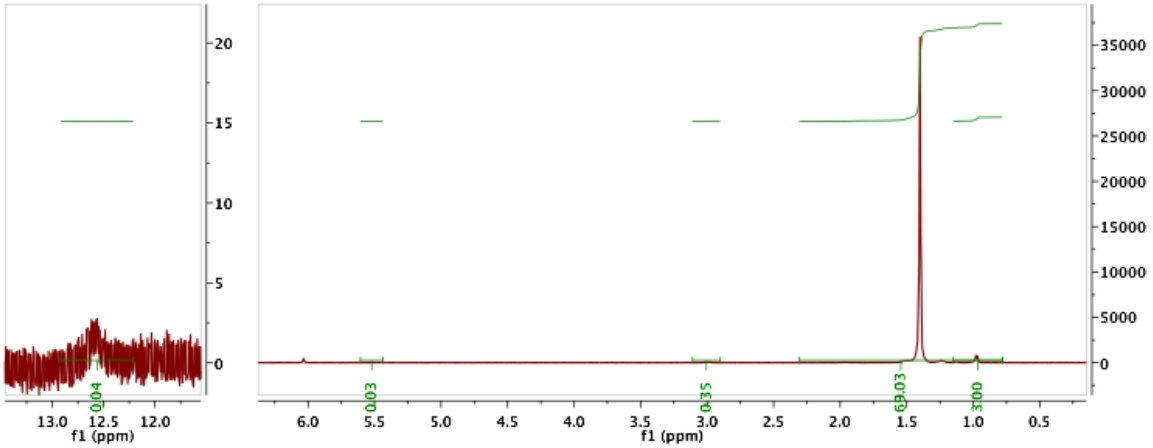
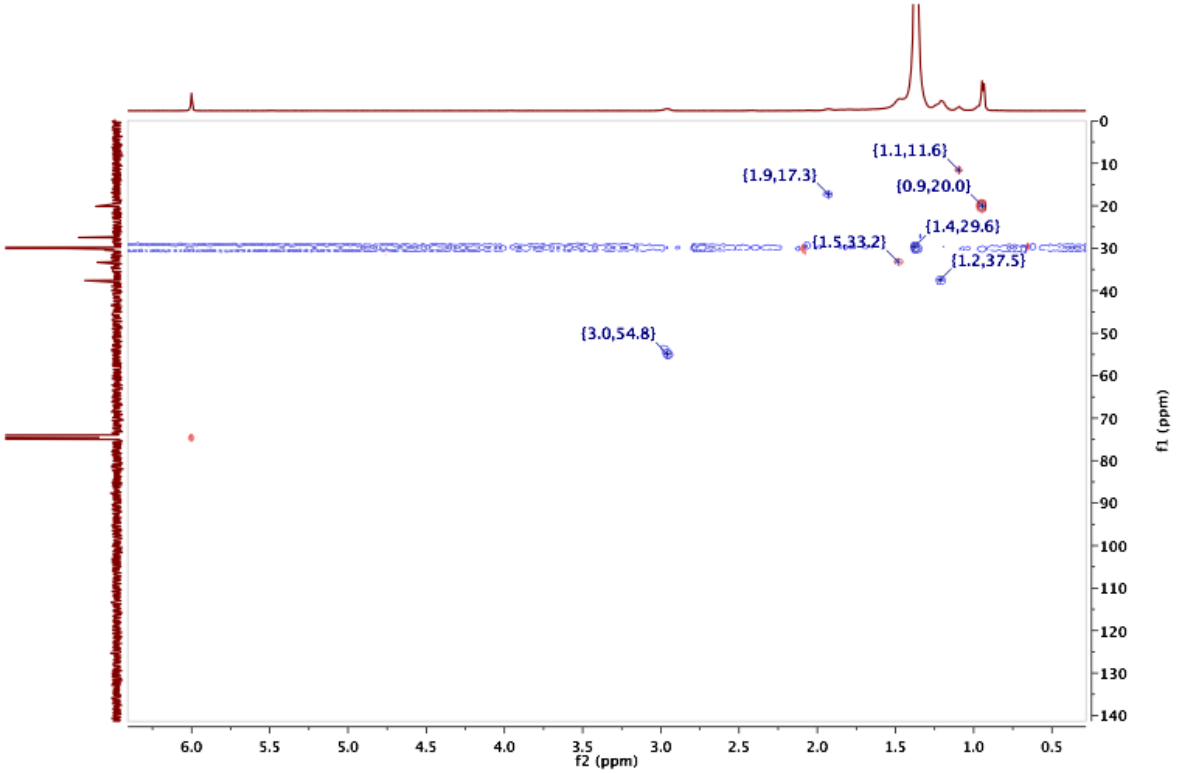


Figure S71. ^{1}H-^{13}C HSQCAD NMR spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **2-s** with the largest cross peaks labeled. The peaks around 55 ppm were too small to observe in the ^{13}C NMR spectrum.



DOSY and TOCSY NMR Experiments

Figure S72. DOSY spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/1-hexene copolymer made with **1-s**. There are no peaks around 3 ppm indicating that no NCH_2 protons are present.

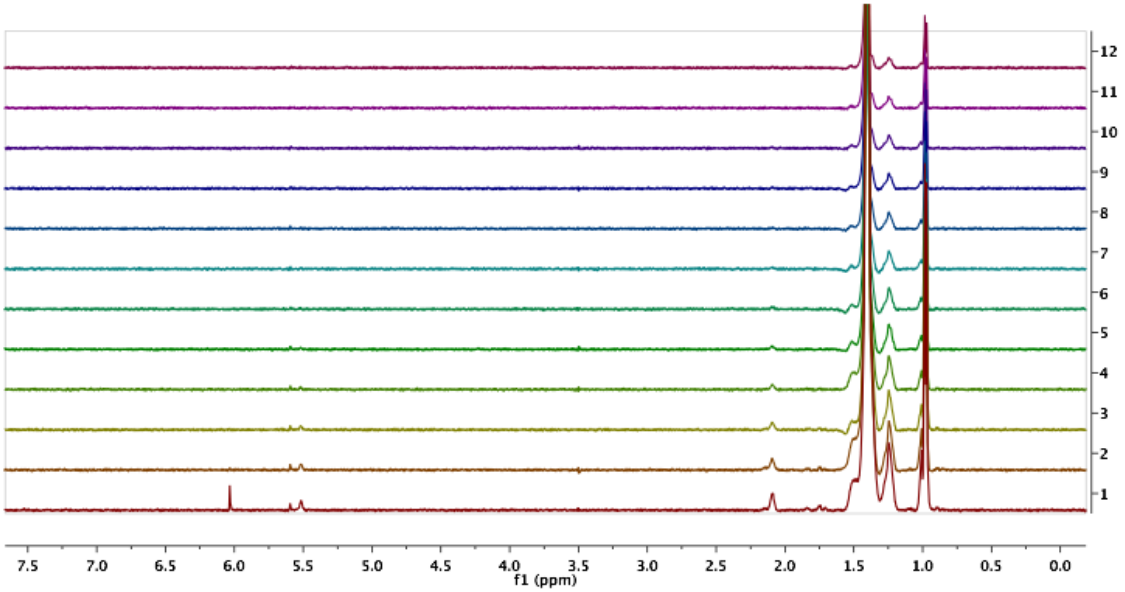


Figure S73. DOSY spectrum in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **1-s**. The peaks at 3 ppm are attenuated at the same rate at the other polymer peaks indicating that the NCH_2 peaks are part of the polymer.

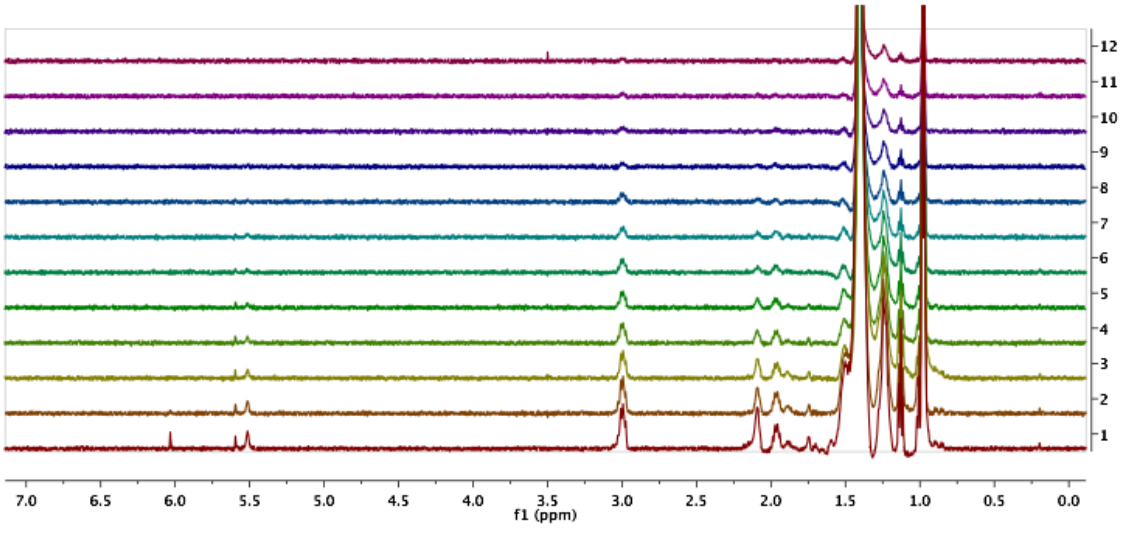
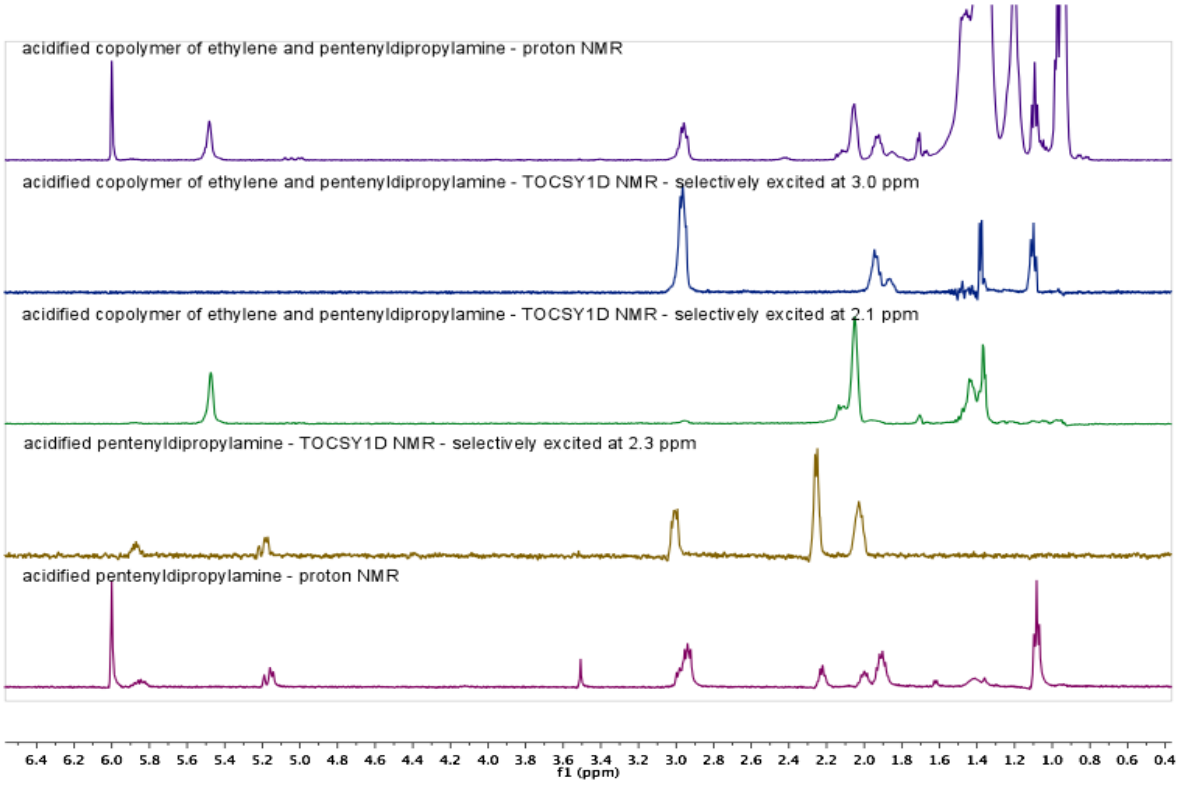


Figure S74. TOCSY spectra in tetrachloroethane-D_2 at 130 °C of the ethylene/N(pentenyl)(^{n}Pr)$_2$ copolymer made with **1-s** and acidified N(pentenyl)(^{n}Pr)$_2$.



Crystallographic Information

Table S5. Crystal and refinement data for complexes **1-s** and **2-s**.

	1-s	**2-s**
empirical formula	$C_{86.50}H_{148}N_2Ni_2O_2P_2$	$C_{86.80}H_{119.20}N_2Ni_2O_2P_2$
formula wt	1427.42	1395.93
T (K)	100	100
a, Å	13.408(2)	13.284(4)
b, Å	14.878(2)	21.951(7)
c, Å	23.068(4)	27.971(9)
α, deg	104.541(8)	91.115(7)
β, deg	90.001(9)	97.107(8)
γ, deg	116.551(8)	101.877(7)
V, Å^3	3950.8(12)	7912(4)
Z	2	4
cryst syst	triclinic	triclinic
space group	P -1	P -1
d_{calcd}, g/cm^3	1.200	1.171
θ range, deg	1.6 to 29.0	1.8 to 27.7
μ, mm^{-1}	0.56	0.56
abs cor	Semi-empirical from equivalents	Semi-empirical from equivalents
GOF	1.77	1.91
R1,[a] wR2[b] (I > 2θ (I))	R1 = 0.1425, wR2 = 0.3256	R1 = 0.1615, wR2 = 0.3432

[a] $R1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$.

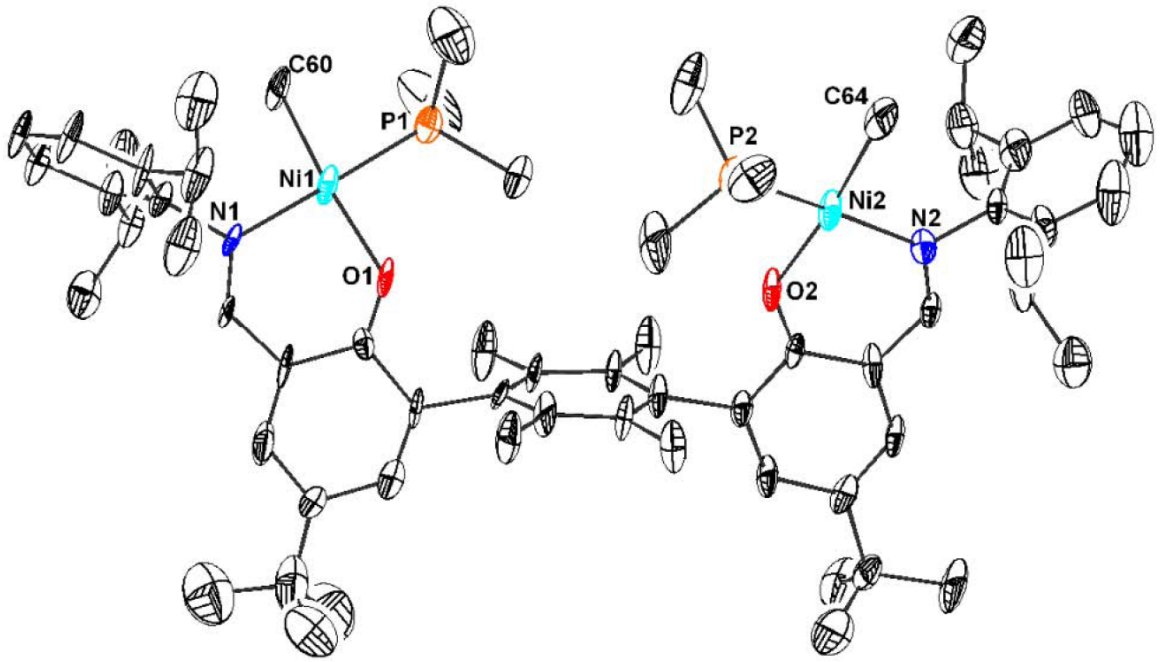


Figure S75. Structural drawing of **1-s**.

Table S6. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **1-s**. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ni(1)	10394(1)	2230(1)	9359(1)	31(1)
Ni(2)	8104(1)	-2219(1)	5650(1)	31(1)
P(1)	10965(2)	2024(2)	8492(1)	52(1)
P(2)	8745(2)	-2188(2)	6505(1)	40(1)
O(1)	8870(5)	1494(4)	8968(2)	31(1)
O(2)	7284(5)	-1550(4)	6049(2)	31(1)
N(1)	9902(6)	2512(4)	10162(2)	19(2)
N(2)	7367(6)	-2484(5)	4859(3)	26(2)
C(1)	6234(8)	-639(7)	7919(3)	43(3)
C(2)	6828(6)	458(6)	8123(3)	25(2)
C(3)	7173(7)	1024(6)	7714(3)	34(2)
C(4)	6929(7)	524(7)	7088(3)	34(2)
C(5)	6277(7)	-539(6)	6882(3)	31(2)
C(6)	5918(7)	-1147(6)	7294(3)	35(2)
C(7)	5852(9)	-1304(7)	8365(4)	54(3)
C(8)	7833(10)	2227(7)	7946(4)	67(3)
C(9)	7353(9)	1185(7)	6659(4)	59(3)
C(10)	5183(9)	-2315(7)	7068(4)	54(3)
C(11)	6915(8)	960(6)	8785(3)	36(2)
C(12)	7984(7)	1435(6)	9172(3)	23(2)
C(13)	7936(8)	1870(7)	9799(3)	41(2)
C(14)	6942(9)	1878(9)	9989(4)	73(4)
C(15)	5973(9)	1542(11)	9602(5)	95(5)
C(16)	6013(9)	1015(9)	8996(4)	66(3)
C(17)	4937(14)	1695(11)	9762(5)	88(4)

C(18)	5115(13)	2354(11)	10390(6)	106(5)
C(19)	4044(16)	660(20)	9776(8)	233(15)
C(20)	4574(15)	2137(16)	9359(6)	146(8)
C(21)	8904(7)	2340(6)	10239(3)	24(2)
C(22)	10708(7)	3045(6)	10706(3)	27(2)
C(23)	11120(7)	4123(6)	10956(3)	32(2)
C(24)	11789(7)	4609(6)	11523(4)	40(2)
C(25)	12079(8)	4038(7)	11795(4)	45(3)
C(26)	11711(8)	2960(7)	11542(3)	48(3)
C(27)	11010(8)	2448(6)	10978(3)	44(3)
C(28)	10804(8)	4781(6)	10656(4)	40(2)
C(29)	10024(8)	5173(7)	11001(4)	50(3)
C(30)	11878(10)	5689(7)	10561(4)	64(3)
C(31)	10590(9)	1284(7)	10698(4)	52(3)
C(32)	9523(10)	677(7)	10938(5)	66(3)
C(33)	11514(10)	948(8)	10773(5)	68(3)
C(34)	5825(7)	-1059(6)	6224(3)	28(2)
C(35)	6408(7)	-1509(6)	5831(3)	23(2)
C(36)	5962(8)	-1933(6)	5211(3)	29(2)
C(37)	4954(7)	-1961(6)	5013(3)	33(2)
C(38)	4354(7)	-1586(6)	5397(3)	31(2)
C(39)	4853(8)	-1102(6)	6000(3)	35(2)
C(40)	3241(8)	-1632(8)	5196(4)	44(3)
C(41)	2395(10)	-2131(10)	5597(5)	80(4)
C(42)	3386(9)	-536(8)	5234(5)	65(3)
C(43)	2746(9)	-2313(8)	4544(4)	63(3)
C(44)	6517(7)	-2350(6)	4767(3)	26(2)
C(45)	7657(7)	-2999(6)	4293(3)	26(2)
C(46)	8507(9)	-2394(8)	4021(4)	45(3)
C(47)	8747(9)	-2915(8)	3488(4)	49(3)
C(48)	8149(11)	-3984(8)	3226(4)	65(3)
C(49)	7278(11)	-4546(8)	3533(4)	68(4)
C(50)	7061(8)	-4080(7)	4066(3)	38(2)
C(51)	9196(8)	-1220(7)	4286(4)	44(3)
C(52)	8664(10)	-690(8)	4030(5)	70(3)
C(53)	10438(9)	-792(8)	4176(4)	58(3)
C(54)	6146(9)	-4763(6)	4386(4)	47(3)
C(55)	4900(9)	-5230(8)	4013(4)	61(3)
C(56)	6360(9)	-5611(6)	4521(4)	59(3)
C(57)	12031(11)	1595(9)	8371(5)	80(4)
C(58)	11516(14)	3226(13)	8262(6)	122(6)
C(59)	9868(10)	1027(12)	7867(5)	115(6)
C(60)	11959(8)	2965(7)	9673(4)	51(3)
C(61)	8415(9)	-3464(8)	6577(5)	65(3)
C(62)	8114(11)	-1732(10)	7130(4)	83(4)
C(63)	10259(9)	-1434(7)	6779(4)	69(4)
C(64)	9110(8)	-2808(8)	5301(4)	51(3)

Table S7. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for **1-s**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	447(8)	322(7)	138(5)	11(4)	-125(5)	186(6)
Ni(2)	504(8)	331(7)	146(5)	56(4)	-78(5)	246(6)
P(1)	448(17)	830(20)	223(12)	-8(12)	-74(11)	321(15)
P(2)	552(17)	541(16)	219(11)	128(11)	-56(11)	349(14)
O(1)	460(40)	380(40)	80(20)	-20(20)	-140(30)	230(30)
O(2)	620(40)	300(30)	90(20)	0(20)	-90(30)	310(30)
N(1)	260(40)	160(40)	130(30)	0(20)	-130(30)	90(30)
N(2)	400(50)	250(40)	200(30)	60(30)	10(30)	200(30)
C(1)	690(70)	610(70)	100(40)	80(40)	-110(40)	410(60)
C(2)	220(50)	260(50)	130(40)	-40(30)	-120(30)	50(40)
C(3)	410(60)	380(50)	80(40)	-30(30)	-110(40)	100(40)
C(4)	520(60)	360(60)	70(40)	0(30)	-80(40)	180(50)
C(5)	590(60)	300(50)	90(40)	40(30)	-80(40)	260(50)
C(6)	550(60)	220(50)	120(40)	-30(30)	-100(40)	90(40)
C(7)	780(80)	500(60)	170(40)	80(40)	-60(50)	150(60)
C(8)	1020(90)	330(60)	330(50)	-160(40)	-150(60)	170(60)
C(9)	1020(90)	390(60)	200(50)	30(40)	-50(50)	230(60)
C(10)	830(80)	400(60)	230(50)	-20(40)	20(50)	210(60)
C(11)	460(60)	350(50)	90(40)	-90(30)	-110(40)	120(50)
C(12)	350(50)	270(50)	130(40)	40(30)	20(40)	200(40)
C(13)	530(70)	450(60)	80(40)	-50(40)	-170(40)	160(50)
C(14)	480(70)	1030(90)	150(50)	-110(50)	-40(50)	30(60)
C(15)	350(70)	1720(130)	330(60)	-330(70)	-140(50)	420(80)
C(16)	450(70)	970(90)	280(50)	-130(50)	-110(50)	230(60)
C(17)	1010(120)	1020(110)	370(70)	-50(70)	-60(70)	380(90)
C(18)	1350(140)	1170(120)	790(100)	290(90)	230(90)	700(110)
C(19)	980(160)	4100(400)	950(150)	-300(200)	320(120)	900(200)
C(20)	1570(160)	2900(200)	690(100)	420(120)	40(100)	1690(170)
C(21)	290(50)	330(50)	60(30)	20(30)	-100(30)	120(40)
C(22)	350(40)	310(40)	160(30)	60(30)	-130(30)	160(30)
C(23)	550(60)	200(50)	180(40)	70(30)	-120(40)	130(40)
C(24)	520(60)	200(50)	260(40)	20(40)	-170(40)	10(40)
C(25)	580(70)	450(60)	170(40)	10(40)	-240(40)	150(50)
C(26)	680(70)	390(60)	180(40)	100(40)	-280(40)	90(50)
C(27)	710(70)	220(50)	190(40)	-80(40)	-290(40)	130(50)
C(28)	570(70)	240(50)	250(40)	20(40)	-160(40)	80(50)
C(29)	730(80)	300(60)	480(60)	-30(40)	-110(50)	310(50)
C(30)	1110(100)	390(70)	360(60)	100(50)	-60(60)	300(70)
C(31)	870(80)	250(60)	390(50)	20(40)	-320(50)	250(60)
C(32)	900(90)	240(60)	540(70)	60(50)	-210(60)	50(60)
C(33)	1070(100)	590(70)	540(70)	230(50)	-190(60)	480(70)
C(34)	420(60)	310(50)	140(40)	40(30)	-50(40)	220(40)
C(35)	340(50)	230(50)	100(40)	60(30)	-60(30)	90(40)
C(36)	670(70)	230(50)	110(40)	40(30)	-30(40)	310(50)
C(37)	530(60)	330(50)	110(40)	10(30)	-110(40)	200(50)
C(38)	430(60)	310(50)	230(40)	100(40)	-120(40)	180(40)
C(39)	590(60)	410(60)	110(40)	50(30)	-50(40)	300(50)
C(40)	410(60)	630(70)	270(50)	20(40)	-120(40)	300(50)

C(41)	690(90)	1240(110)	500(70)	190(70)	-80(60)	490(80)
C(42)	840(90)	620(80)	620(70)	0(50)	-240(60)	530(70)
C(43)	910(90)	720(80)	270(50)	-20(50)	-300(50)	480(70)
C(44)	410(60)	260(50)	130(40)	30(30)	-60(40)	180(40)
C(45)	400(50)	260(50)	130(40)	30(30)	-20(40)	170(40)
C(46)	650(70)	630(70)	270(50)	160(50)	-10(50)	450(60)
C(47)	750(80)	580(70)	220(50)	220(50)	190(50)	310(60)
C(48)	1350(110)	360(70)	370(60)	90(50)	0(70)	520(70)
C(49)	1310(110)	330(60)	410(60)	20(50)	-90(70)	430(70)
C(50)	660(70)	460(60)	210(40)	40(40)	40(40)	440(60)
C(51)	670(80)	310(60)	340(50)	80(40)	80(50)	230(50)
C(52)	1000(100)	440(70)	560(70)	140(50)	120(70)	270(70)
C(53)	590(80)	460(70)	500(60)	110(50)	-80(50)	90(60)
C(54)	780(80)	200(50)	270(50)	-50(40)	30(50)	150(50)
C(55)	780(80)	470(70)	500(60)	80(50)	50(60)	240(60)
C(56)	1080(90)	110(50)	480(60)	180(40)	110(60)	140(50)
C(57)	1220(110)	680(80)	750(80)	180(60)	280(80)	660(80)
C(58)	1990(170)	2260(170)	790(90)	1150(110)	820(100)	1770(150)
C(59)	550(80)	1950(150)	350(70)	-350(80)	-180(60)	410(90)
C(60)	560(70)	650(70)	180(40)	-10(40)	-180(40)	240(50)
C(61)	660(80)	720(80)	660(70)	350(60)	-90(60)	320(60)
C(62)	1430(120)	1230(100)	240(50)	70(60)	-140(60)	1030(100)
C(63)	1020(100)	390(60)	440(60)	100(50)	-300(60)	150(60)
C(64)	600(70)	770(70)	290(50)	150(50)	-140(50)	420(60)

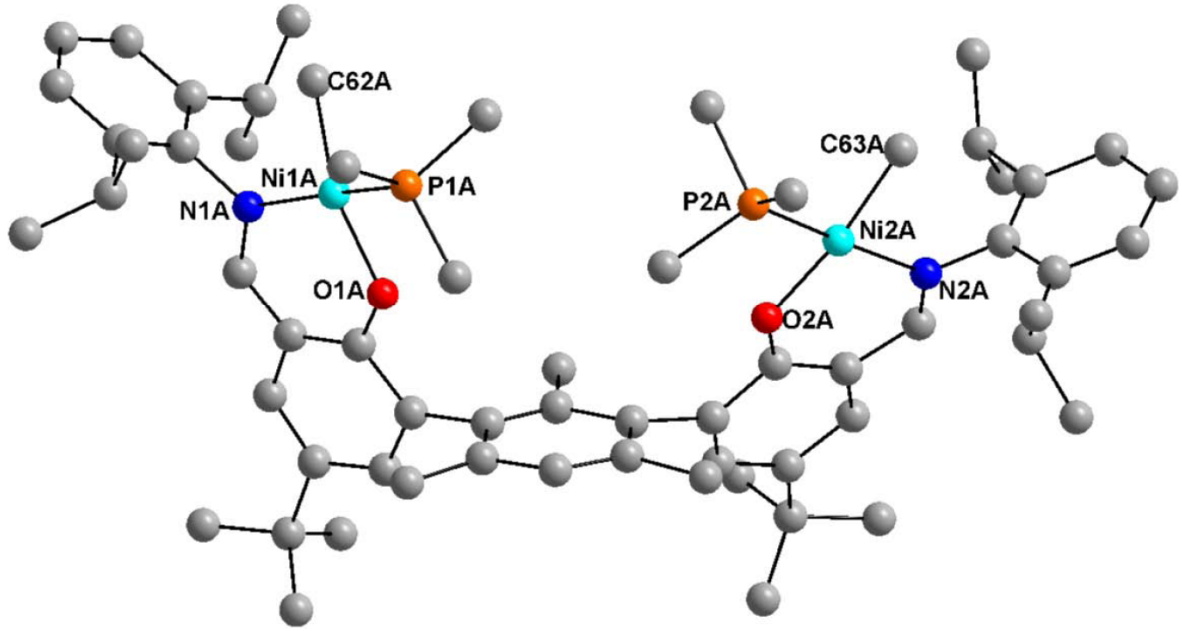


Figure S76. Structural drawing of **2-s**.

Table S8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2-s**. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ni(1A)	8155(1)	4620(1)	8638(1)	16(1)
Ni(2A)	8925(1)	890(1)	8733(1)	19(1)
P(1A)	9528(3)	4295(2)	8858(1)	22(1)
P(2A)	10057(3)	1735(2)	8837(1)	27(1)
O(1A)	7453(7)	4081(4)	9070(3)	14(2)
O(2A)	8017(7)	1241(4)	9065(3)	16(2)
N(1A)	7115(9)	5100(5)	8541(4)	21(3)
N(2A)	7995(9)	62(4)	8655(4)	13(3)
C(1A)	6368(10)	2522(5)	9401(4)	11(3)
C(2A)	6664(10)	3079(6)	9629(4)	14(3)
C(3A)	7395(10)	3214(5)	10026(4)	8(3)
C(4A)	7928(10)	2737(5)	10170(4)	12(3)
C(5A)	7641(11)	2155(6)	9957(5)	19(3)
C(6A)	6869(10)	2021(5)	9556(4)	9(3)
C(7A)	5559(11)	2401(6)	8971(4)	17(3)
C(8A)	7675(12)	3810(6)	10302(5)	34(4)
C(9A)	8166(10)	1630(5)	10157(4)	15(3)
C(10A)	6062(10)	3572(5)	9476(4)	9(3)
C(11A)	6523(10)	4083(5)	9195(4)	9(3)
C(12A)	5921(10)	4514(5)	9042(4)	9(3)
C(13A)	4994(10)	4484(5)	9219(4)	10(3)
C(14A)	4582(10)	4042(5)	9510(4)	8(3)
C(15A)	5127(10)	3585(5)	9633(4)	9(3)
C(16A)	3562(11)	4034(6)	9743(5)	19(3)
C(17A)	3174(12)	4623(6)	9662(5)	26(4)
C(18A)	2734(11)	3510(6)	9510(5)	25(4)
C(19A)	3793(11)	3964(6)	10275(4)	21(3)
C(20A)	6280(12)	4993(6)	8763(5)	20(3)
C(21A)	7180(11)	5645(6)	8226(5)	17(3)
C(22A)	6614(11)	5526(6)	7774(5)	21(3)
C(23A)	6629(12)	6048(6)	7476(5)	29(4)
C(24A)	7197(13)	6635(7)	7640(5)	37(4)
C(25A)	7784(13)	6731(7)	8081(5)	37(4)
C(26A)	7794(11)	6224(6)	8384(5)	21(3)
C(27A)	5976(11)	4905(6)	7592(5)	18(3)
C(28A)	6163(13)	4725(7)	7111(5)	35(4)
C(29A)	4806(11)	4829(6)	7608(5)	29(4)
C(30A)	8451(13)	6362(7)	8877(5)	34(4)
C(31A)	9611(15)	6412(8)	8834(6)	62(6)
C(32A)	8263(13)	6950(7)	9152(5)	41(5)
C(33A)	6429(10)	1343(5)	9365(4)	10(3)
C(34A)	7061(10)	1001(6)	9122(4)	11(3)
C(35A)	6573(10)	384(6)	8989(4)	13(3)
C(36A)	5527(10)	141(6)	9034(4)	12(3)
C(37A)	4918(10)	504(6)	9256(4)	11(3)
C(38A)	5460(10)	1117(5)	9417(4)	7(3)
C(39A)	3758(11)	272(6)	9299(5)	17(3)
C(40A)	3510(16)	387(8)	9786(6)	66(6)

C(41A)	3143(15)	598(8)	8941(6)	59(6)
C(42A)	3416(13)	-455(6)	9168(5)	38(4)
C(43A)	7095(11)	-57(6)	8770(5)	18(3)
C(44A)	8270(12)	-461(6)	8456(5)	24(4)
C(45A)	7963(13)	-672(7)	7989(6)	38(4)
C(46A)	8332(13)	-1211(7)	7813(6)	37(4)
C(47A)	8894(16)	-1525(9)	8131(7)	68(6)
C(48A)	9172(13)	-1301(7)	8581(6)	38(4)
C(49A)	8913(11)	-765(6)	8768(5)	21(3)
C(50A)	7347(14)	-364(8)	7644(6)	44(5)
C(51A)	6225(17)	-740(9)	7578(7)	84(7)
C(52A)	7765(15)	-194(8)	7185(6)	63(6)
C(53A)	9255(12)	-565(7)	9288(5)	30(4)
C(54A)	8781(17)	-1045(9)	9642(7)	85(7)
C(55A)	10517(13)	-402(7)	9436(6)	46(5)
C(56A)	10608(12)	4950(6)	9044(5)	33(4)
C(57A)	10004(12)	3818(6)	8467(5)	31(4)
C(58A)	9502(13)	3864(7)	9392(5)	37(4)
C(59A)	9818(15)	2332(8)	9243(6)	62(6)
C(60A)	10255(13)	2176(7)	8306(5)	36(4)
C(61A)	11397(12)	1664(7)	9065(5)	33(4)
C(62A)	8854(11)	4953(6)	8107(4)	17(3)
C(63A)	9881(12)	625(6)	8342(5)	29(4)
Ni(1B)	8750(2)	6655(1)	3267(1)	29(1)
Ni(2B)	9239(2)	2812(1)	3201(1)	29(1)
P(1B)	10142(4)	6337(2)	3468(2)	32(1)
P(2B)	10390(4)	3643(2)	3350(2)	32(1)
O(1B)	8052(8)	6076(4)	3680(3)	32(3)
O(2B)	8338(9)	3161(5)	3541(4)	37(3)
N(1B)	7650(10)	7137(5)	3166(4)	28(3)
N(2B)	8266(11)	2035(6)	3057(4)	37(3)
C(1B)	6894(12)	4395(7)	3945(5)	29(4)
C(2B)	7213(12)	4998(7)	4202(5)	30(4)
C(3B)	8051(13)	5077(7)	4596(5)	32(4)
C(4B)	8389(13)	4568(7)	4727(5)	35(4)
C(5B)	8086(12)	3989(7)	4493(5)	29(4)
C(6B)	7322(12)	3906(7)	4104(5)	29(4)
C(7B)	6040(13)	4329(7)	3522(5)	39(4)
C(8B)	8352(14)	5709(7)	4858(6)	49(5)
C(9B)	8497(13)	3411(7)	4684(6)	43(5)
C(10B)	6728(12)	5519(6)	4062(5)	24(4)
C(11B)	7124(13)	6017(7)	3816(5)	30(4)
C(12B)	6543(12)	6510(7)	3695(5)	29(4)
C(13B)	5581(13)	6460(7)	3876(5)	35(4)
C(14B)	5208(14)	5964(7)	4145(6)	39(4)
C(15B)	5811(13)	5535(7)	4230(5)	35(4)
C(16B)	4201(14)	5932(7)	4382(6)	39(4)
C(17B)	3630(18)	6478(10)	4253(8)	91(8)
C(18B)	3422(14)	5292(8)	4259(6)	55(5)
C(19B)	4443(14)	5986(8)	4925(6)	50(5)
C(20B)	6845(13)	7003(7)	3382(5)	34(4)
C(21B)	7684(11)	7630(6)	2857(5)	20(3)
C(22B)	7145(12)	7537(6)	2393(5)	25(4)
C(23B)	7111(12)	8045(6)	2117(5)	28(4)

C(24B)	7695(13)	8616(7)	2279(5)	35(4)
C(25B)	8239(13)	8698(7)	2718(5)	36(4)
C(26B)	8264(13)	8216(7)	3037(6)	39(4)
C(27B)	6518(11)	6906(6)	2229(5)	22(4)
C(28B)	6683(13)	6739(7)	1744(5)	41(4)
C(29B)	5363(12)	6877(7)	2232(5)	36(4)
C(30B)	8850(16)	8323(9)	3527(7)	59(6)
C(31B)	9952(19)	8721(10)	3539(8)	107(9)
C(32B)	8285(17)	8534(9)	3916(7)	82(7)
C(33B)	6851(11)	3285(6)	3898(5)	21(4)
C(34B)	7381(12)	2918(6)	3633(5)	23(4)
C(35B)	6867(12)	2317(7)	3467(5)	28(4)
C(36B)	5891(16)	2068(9)	3553(6)	58(5)
C(37B)	5381(14)	2393(8)	3814(6)	43(5)
C(38B)	5823(14)	2990(7)	3981(6)	43(5)
C(39B)	4212(16)	2123(9)	3876(7)	58(6)
C(40B)	4240(30)	2214(16)	4434(13)	219(18)
C(41B)	3560(30)	2504(17)	3728(13)	228(19)
C(42B)	3925(18)	1439(9)	3853(8)	91(8)
C(43B)	7345(14)	1916(8)	3220(6)	47(5)
C(44B)	8483(14)	1475(7)	2801(6)	41(5)
C(45B)	8226(13)	1418(7)	2329(5)	33(4)
C(46B)	8406(16)	902(9)	2088(7)	68(6)
C(47B)	8937(15)	494(9)	2344(7)	63(6)
C(48B)	9293(15)	597(8)	2810(6)	51(5)
C(49B)	9060(14)	1081(8)	3085(6)	49(5)
C(50B)	7683(14)	1862(8)	2053(6)	49(5)
C(51B)	6488(15)	1609(9)	1987(7)	70(6)
C(52B)	8029(15)	1980(8)	1558(6)	61(6)
C(53B)	9402(14)	1198(8)	3620(6)	51(5)
C(54B)	8736(18)	719(10)	3889(8)	90(7)
C(55B)	10497(17)	1130(10)	3753(8)	88(7)
C(56B)	11243(14)	6980(7)	3672(6)	52(5)
C(57B)	10681(13)	5912(7)	3036(5)	37(4)
C(58B)	10200(14)	5884(7)	3991(6)	46(5)
C(59B)	10136(14)	4210(7)	3757(6)	50(5)
C(60B)	10679(13)	4127(7)	2850(5)	46(5)
C(61B)	11646(14)	3540(8)	3628(6)	55(5)
C(62B)	9494(12)	7078(7)	2767(5)	37(4)
C(63B)	10203(13)	2541(7)	2813(6)	43(5)
C(1)	7457(17)	2036(9)	7703(7)	67(6)
C(2)	7508(14)	2300(8)	8177(6)	49(5)
C(3)	7390(13)	2898(7)	8235(6)	42(5)
C(4)	7267(15)	3228(9)	7838(6)	56(5)
C(5)	7207(14)	2986(8)	7392(6)	50(5)
C(6)	7256(15)	2428(9)	7300(7)	63(6)
C(7)	7540(30)	1425(14)	7576(11)	169(13)
C(8)	7799(13)	4128(7)	2238(6)	34(4)
C(9)	7931(13)	4360(7)	2706(6)	39(4)
C(10)	7927(14)	4962(8)	2812(7)	57(5)
C(11)	7820(13)	5362(8)	2437(6)	41(5)
C(12)	7673(14)	5123(8)	1971(6)	49(5)
C(13)	7680(15)	4459(9)	1863(7)	64(6)
C(14)	7745(17)	3430(9)	2149(7)	83(7)

C(15)	4870(14)	2919(7)	1302(6)	41(5)
C(16)	4058(15)	2446(8)	1421(6)	52(5)
C(17)	3895(16)	1872(8)	1169(6)	57(5)
C(18)	4445(15)	1734(9)	832(7)	60(6)
C(19)	5252(13)	2214(7)	704(6)	41(4)
C(20)	5442(14)	2800(7)	956(6)	43(5)
C(21)	5053(15)	3519(7)	1539(6)	55(5)
C(22)	5440(20)	8875(13)	2950(10)	82(9)
C(23)	4990(30)	8762(16)	2470(12)	123(13)
C(24)	5020(20)	9137(12)	2092(10)	77(9)
C(25)	5700(20)	9799(14)	2253(10)	95(10)
C(26)	6240(20)	9914(12)	2740(9)	76(9)
C(27)	6000(20)	9501(12)	3051(10)	76(9)
C(28)	5380(30)	8407(16)	3270(12)	143(15)

Table S9. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for **2-s**. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1A)	75(8)	143(7)	303(8)	-11(6)	37(7)	78(6)
Ni(2A)	120(8)	157(8)	305(8)	-24(6)	46(7)	81(6)
P(1A)	189(13)	218(12)	262(13)	6(9)	28(9)	74(9)
P(2A)	250(13)	255(13)	305(13)	-2(9)	46(9)	66(9)
O(1A)	140(20)	140(20)	140(20)	13(10)	17(10)	29(11)
O(2A)	160(20)	160(20)	170(20)	9(10)	23(10)	39(11)
N(1A)	210(30)	210(30)	220(30)	12(10)	27(10)	43(12)
N(2A)	130(30)	130(30)	130(30)	4(10)	20(10)	28(11)
Ni(1B)	237(9)	322(9)	326(9)	0(7)	22(7)	92(7)
Ni(2B)	226(9)	312(9)	352(9)	-33(7)	51(7)	95(7)
P(1B)	306(14)	334(13)	331(14)	12(9)	28(9)	80(9)
P(2B)	299(14)	332(13)	333(14)	-7(9)	48(9)	78(9)
O(1B)	320(30)	330(30)	320(30)	13(10)	45(10)	68(11)
O(2B)	370(30)	370(30)	370(30)	13(10)	53(10)	78(11)
N(1B)	280(30)	280(30)	280(30)	11(10)	35(11)	64(12)
N(2B)	360(40)	370(40)	360(40)	13(10)	49(11)	75(12)

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